X-ray Optics Development at NASA MSFC

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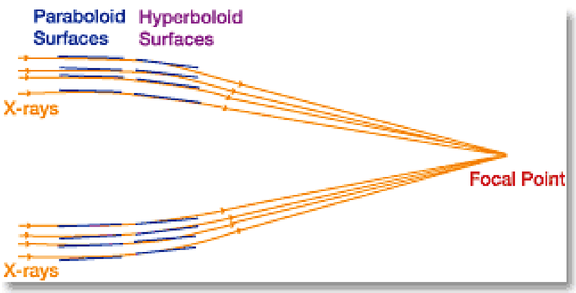
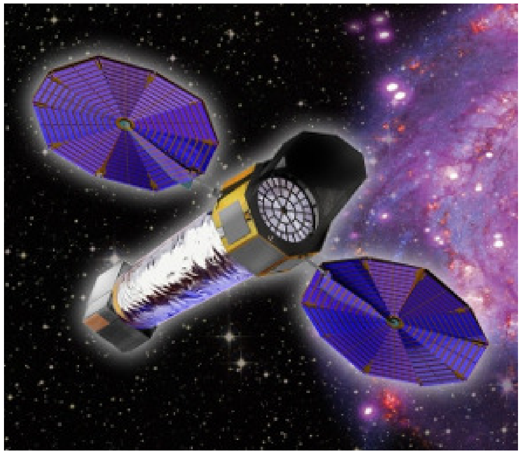
Talk Outline

- X-ray optics for space-borne applications
- Electroformed NiCo X-ray optics at MSFC
- Thin film coatings to enhance X-ray optical performance
- Thin film stress
- In-situ film stress measurement at MSFC: from prototype to refined design
- Ultra lightweight aerogel mirrors

X-ray optics for space-borne applications (Wolter I)



Segments assembled into full shells

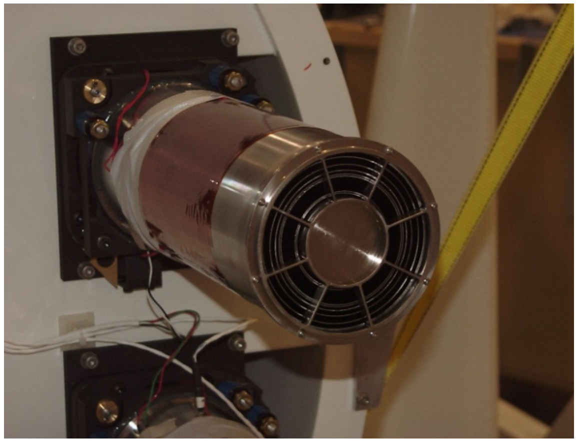


Lynx will require 37,492 segments!
Segments are only ~400 µm thick!

Electroformed X-ray optics at MSFC

Down to 50µm thick

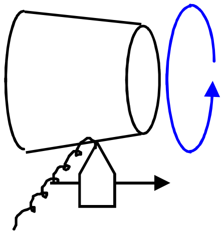
Up to 0.5 m diameter



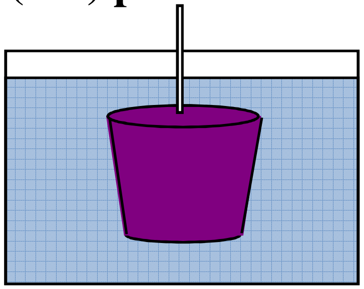
Down to 0.025 m diameter

Electroforming replication process at MSFC

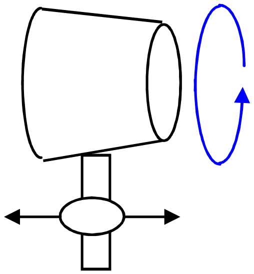
1. CNC Machine, Mandrel Formation From Al Bar



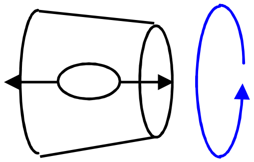
2. Chemical Clean and Activation & Electroless Nickel (EN) plate



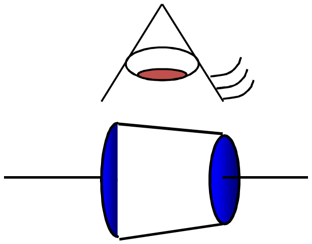
3. Precision Turn to 600Å, sub-micron figure accuracy



4. Polish and Superpolish to 3 - 4Å rms finish

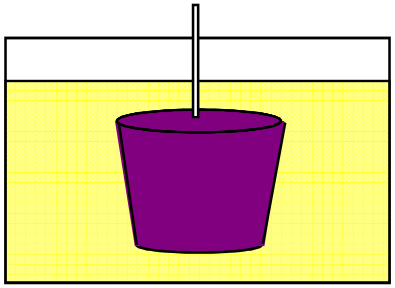


5. Metrology On Mandrel

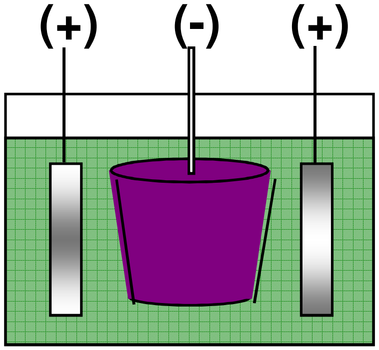


Shell Fabrication

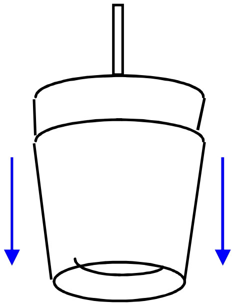
6. Ultrasonic clean and Passivation to Remove Surface Contaminants



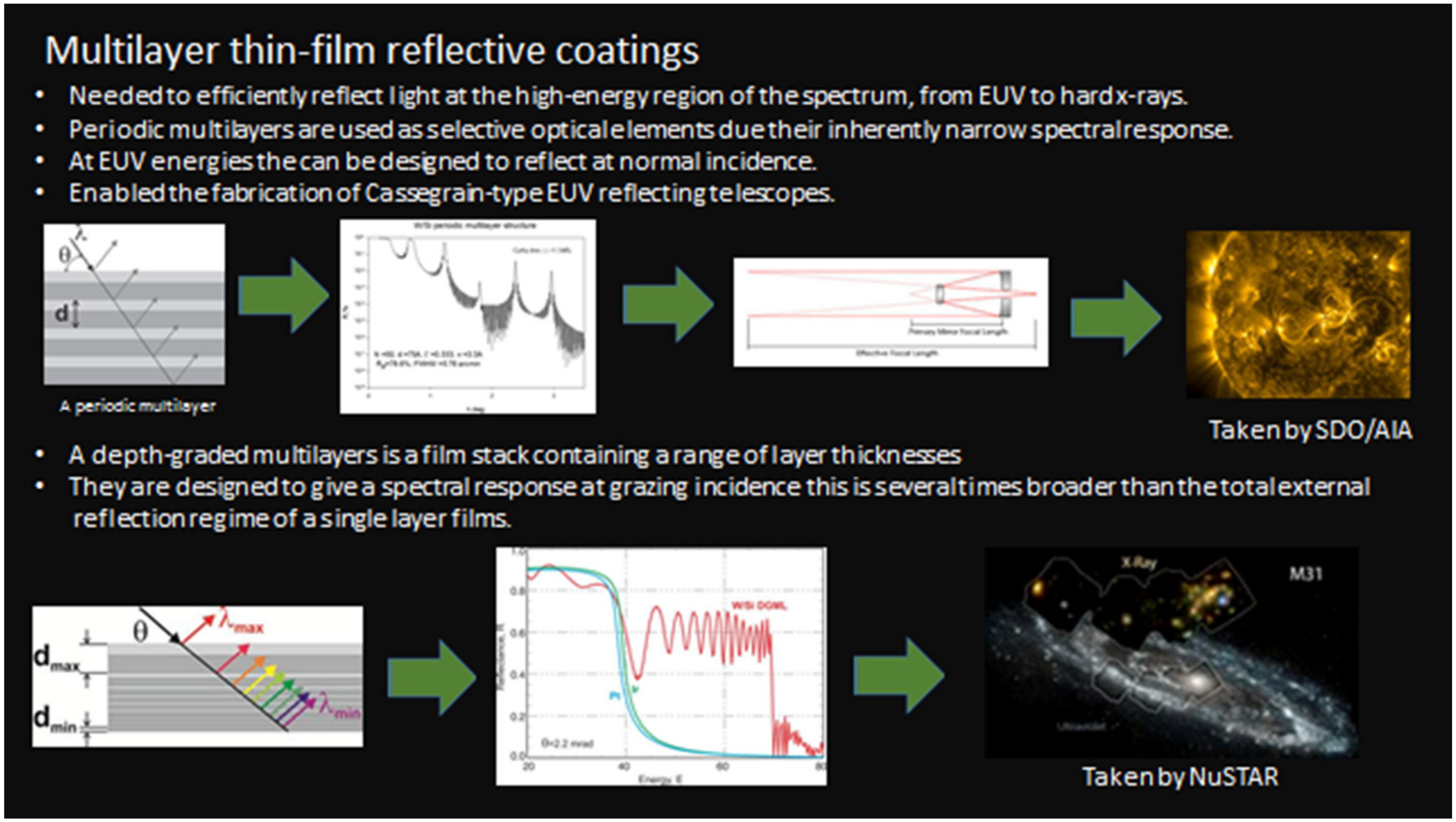
7. Electroform Ni/Co Shell onto Mandrel



8. Separate Optic From Mandrel in Cold Water Bath



Thin film coatings to enhance X-ray reflectivity



Multilayer thin-film reflective coatings

- Needed to efficiently reflect light at the high-energy region of the spectrum, from EUV to hard x-rays.
- Periodic multilayers are used as selective optical elements due their inherently narrow spectral response.
- At EUV energies the can be designed to reflect at normal incidence.
- Enabled the fabrication of Cassegrain-type EUV reflecting telescopes.

A periodic multilayer

Taken by SDO/AIA

- A depth-graded multilayers is a film stack containing a range of layer thicknesses
- They are designed to give a spectral response at grazing incidence this is several times broader than the total external reflection regime of a single layer films.

Taken by NuSTAR

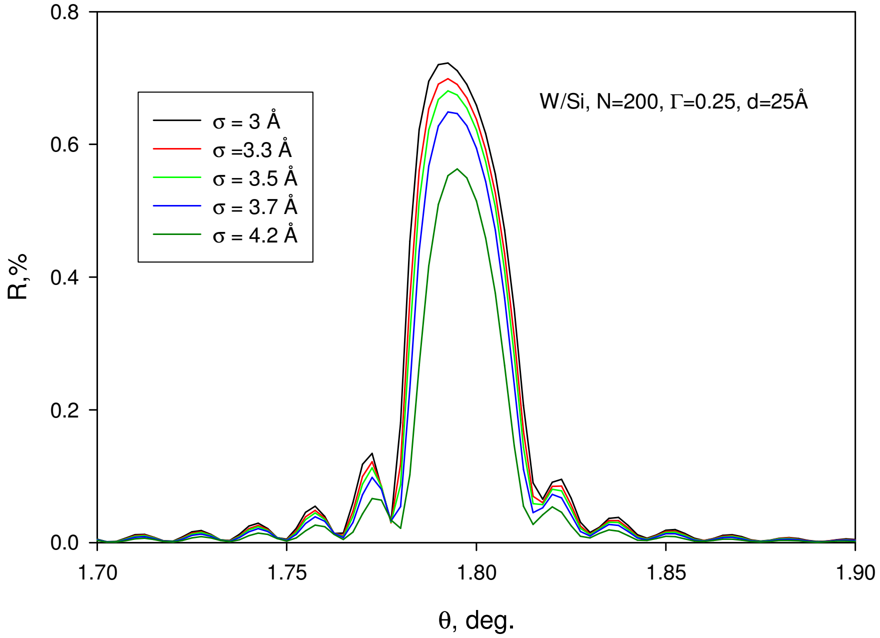
The stress in these coatings will severely deform thin, precisely figured substrates and degrade imaging quality

X-ray reflectivity from the multilayer

Modified Fresnel coefficient:

$$r = r_0 e^{\frac{-8\pi^2\sigma^2}{\lambda^2}\sin(\theta_0)^2}$$

Roughness influence on first order reflectivity



$$r^s_{N-j} = \frac{\sqrt{n^2_{N-j}(\lambda) - \cos(\theta_0)^2} - \sqrt{n^2_{N-j+1}(\lambda) - \cos(\theta_0)^2}}{\sqrt{n^2_{N-j}(\lambda) - \cos(\theta_0)^2} + \sqrt{n^2_{N-j+1}(\lambda) - \cos(\theta_0)^2}}$$

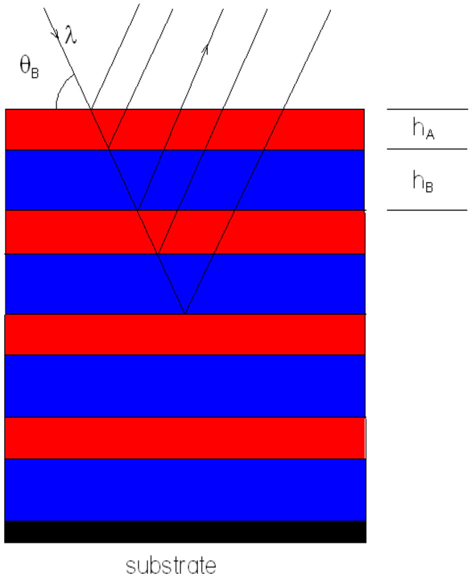
$$r^p_{N-j} = \frac{\left(\frac{n_{N-j}(\lambda)}{n_{N-j+1}(\lambda)}\right)\sqrt{n^2_{N-j+1}(\lambda) - \cos(\theta_0)^2} - \sqrt{n^2_{N-j}(\lambda) - \cos(\theta_0)^2}}{\left(\frac{n_{N-j}(\lambda)}{n_{N-j+1}(\lambda)}\right)\sqrt{n^2_{N-j+1}(\lambda) - \cos(\theta_0)^2} + \sqrt{n^2_{N-j}(\lambda) - \cos(\theta_0)^2}}$$

$$\beta_{N-j} = \frac{2\pi h_{N-j}}{\lambda}\sqrt{1 - \frac{\cos(\theta_0)^2}{n^2_{N-j+1}(\lambda)}}$$

$$S^{\nu}_{j+1} = \frac{r^{\nu}_{N-j} + S^{\nu}_{j} e^{2i\beta_{N-j}}}{1 + r^{\nu}_{N-j} S^{\nu}_{j} e^{2i\beta_{N-j}}}$$

$$R = \frac{1}{2}R^s + \frac{1}{2}R^p$$

$$R^{\nu} = S^{\nu} S^{\nu *}$$



$d \equiv h_A + h_B$

Thin Film Stress

- The stress can be compressive or tensile.

- Various components of stress:
  - Intrinsic, σ_i, which is related to the film's microstructure.
  - Thermal stress, $\sigma_{\Delta CTE}$,which arises due to the difference in the linear expansion coefficient between the film and substrate and the difference between substrate temperature, T_s, during deposition and subsequent cooling to room temperature: $\boxed{\sigma_{\Delta CTE} = M_f(\alpha_s - \alpha_f)\Delta T}$
  - Extrinsic, σ_{ext}, that results due to external forces applied to the film substrate system such as bending of the substrate to produce a figured optic.

- The film stress can be enormous for some materials (i.e. GPa's)

- The curvature,κ, of the deformed substrate is proportional to the product of film stress and film thickness,σh_f, through a constant that describes the geometric and mechanical properties of the substrate (Stoney's Equation)--namely, the substrate's thickness, h_s, and biaxial modulus, $\frac{E_S}{(1-\vartheta_s)}$:

$$\boxed{\sigma h_f = \frac{E_s h_s^2}{6(1-\vartheta s)}\kappa}$$

N/m

Thin Film Stress (Cont’d))

- In controlling film stress, the aim is to manipulate the energy of the sputtered atoms and influence the adatom mobility at the film surface.

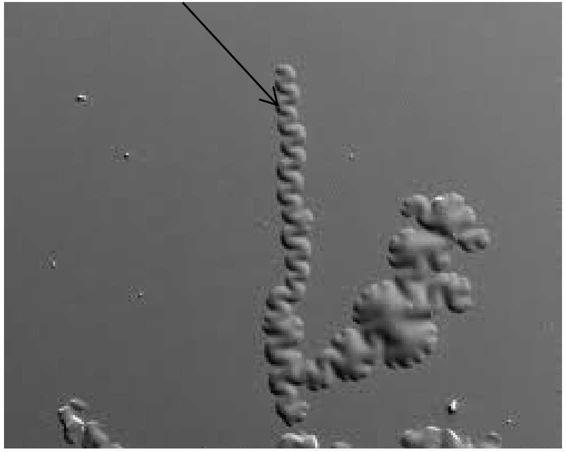
- For a given material, stress is highly process dependent for magnetron sputtering and influenced by deposition conditions:
  - Gas Pressure (stress reversal)
  - Deposition Rate (cathode power)
  - Substrate Temperature
  - Substrate bias

- There is a trade-off between film stress and film quality (i.e. roughness, density).
  - Generally, the deposition conditions needed to achieve good X-ray reflectivity result in high film stress.

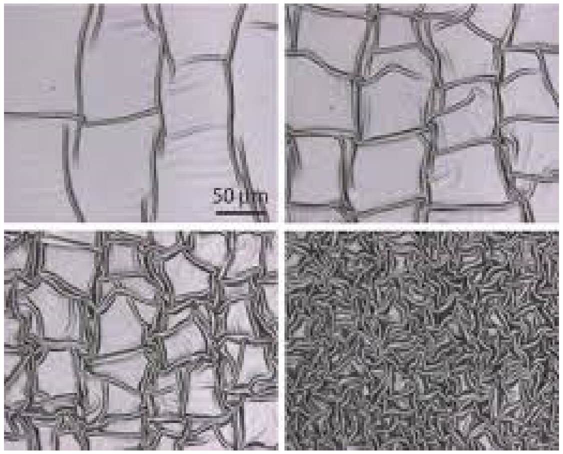
Detrimental effects of high film stress:

- Cracking, buckling and delamination
  If the force per unit length due to the stress in the film exceeds the adhesive force, delamination of the film will occur.

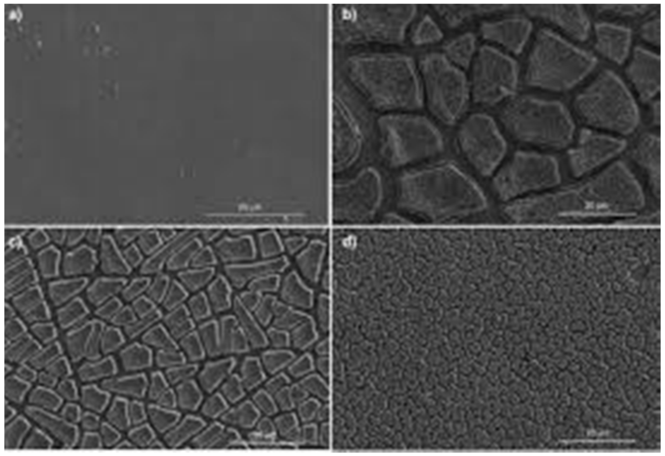
“telephone chord” propagation



High compressive stress (buckling)

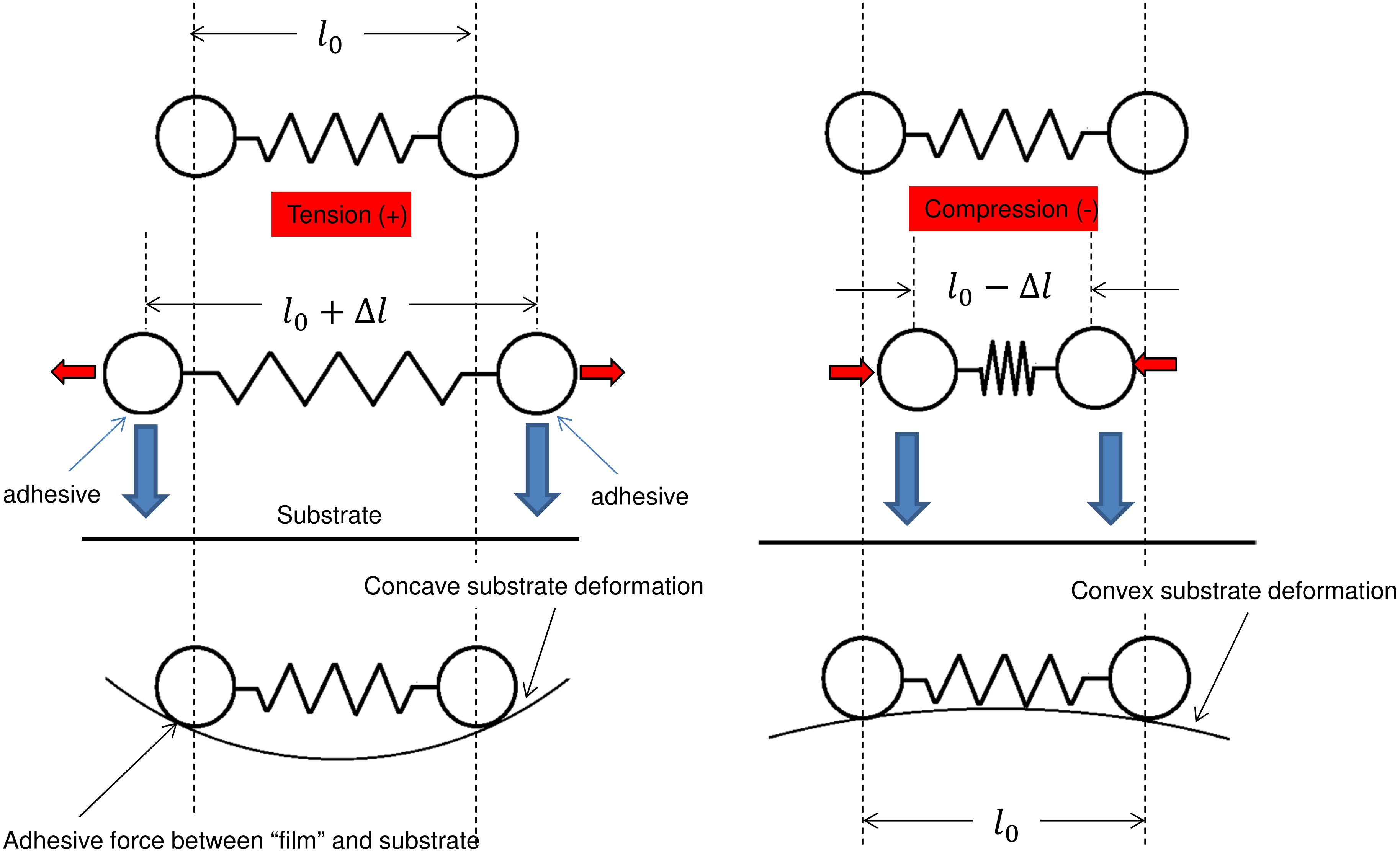


High tensile stress (cracking)



- Substrate deformation
  - Of particular concern for grazing incidence X-ray optics since the stress can alter the precise geometrical figure and degrade its focusing or collimating properties.
    - Significant technological challenge for the next generation of lightweight X-ray space telescopes like Lynx:
    - The desire to achieve sub-second resolution has motivated deposition techniques to correct substrate figure errors which rely on a very low stress film (ie. A few MPa)
    - Substrates are only 10’s of microns thick.
    - The X-ray reflective Ir layer is highly stressed (~4 GPa)

Stress/Spring Model



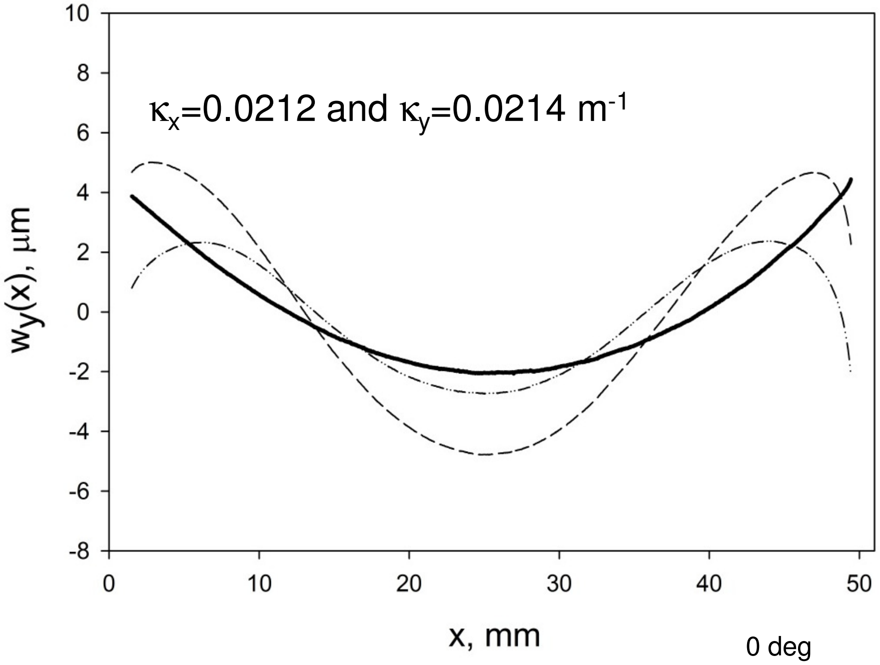
The film will delaminate if the stress is greater than the adhesion between the film as substrate

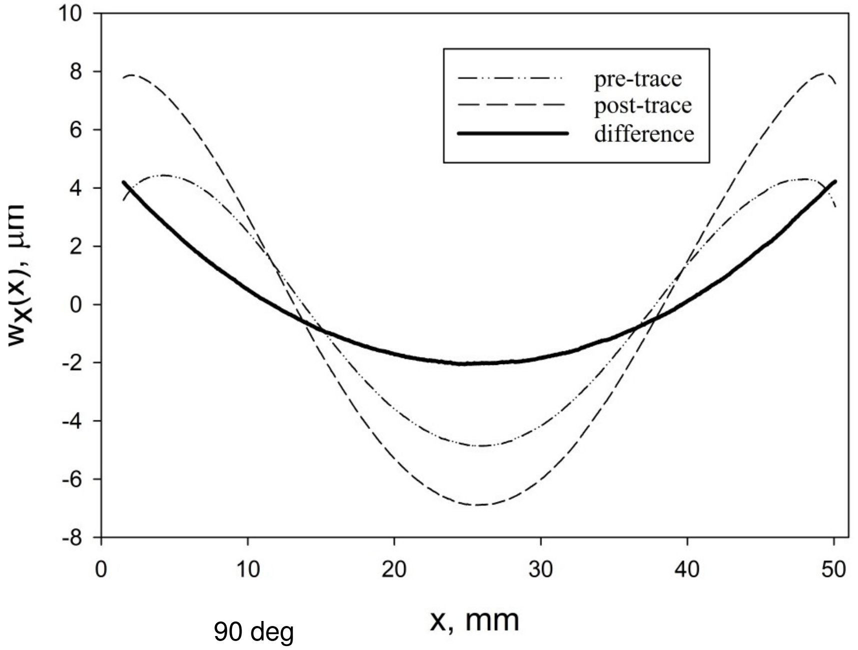
Measurement of thin film stress (ex-situ)

$$Stoney's\ Eqn:\ \sigma h_f = \frac{E_s h_s^2}{6(1-\vartheta s)}\kappa$$

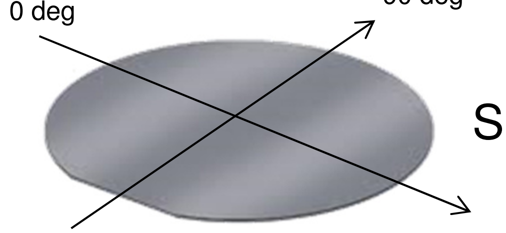
$$Spherical\ Deformation\ Mode:$$
$$A = \sigma h_f \frac{D_s^2}{h_s^3}$$

Tallysurf stylus profilometer





$$\kappa \approx \frac{d^2 w}{d^2 x} = const$$

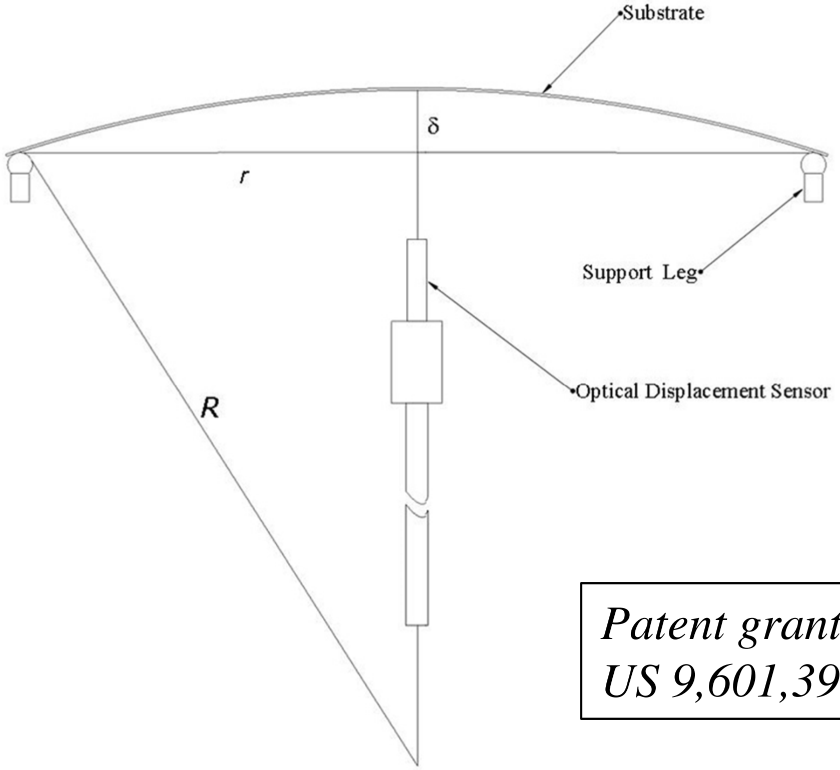


Si <111>

MSFC In-Situ stress measurement methodology:

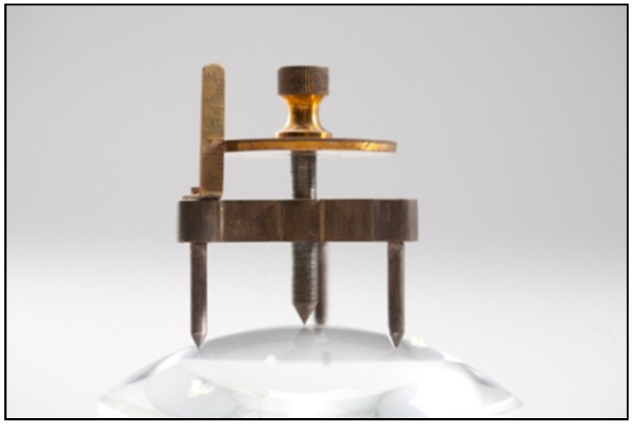
Since substrate deformation is spherical we need only measure the sag, δ, to infer its curvature.

$$\sigma h_f = \frac{E_s h_s^2}{6(1-\vartheta s)}\kappa \quad \text{,where} \quad \kappa = \frac{2\delta}{r^2+\delta^2} \quad \xRightarrow{r \approx \frac{D_s}{2} \gg \delta} \quad \boxed{\sigma h_f = \frac{4}{3}\frac{E_s}{(1-\vartheta s)}\left(\frac{h_s}{D_s}\right)^2 \delta}$$

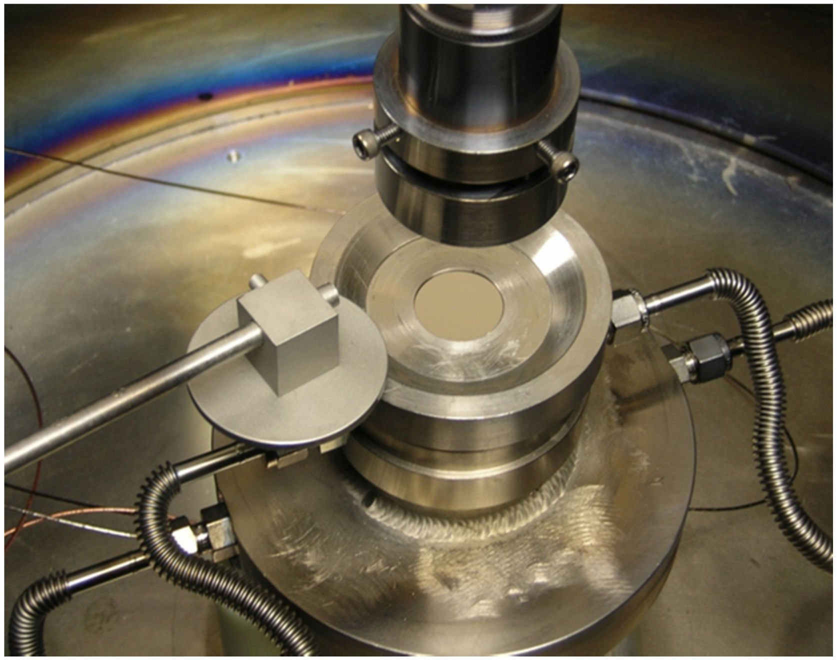


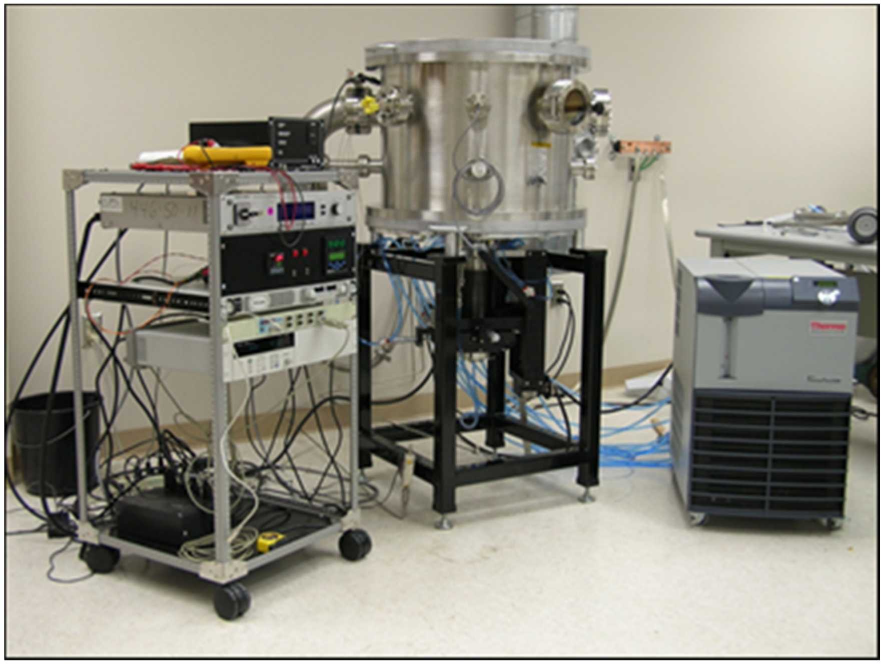
The curvature measurement is performed during deposition by measuring the backside of a double side polished substrate with a non-contact variation of the classic spherometer using a high resolution fiber optic displacement sensor.

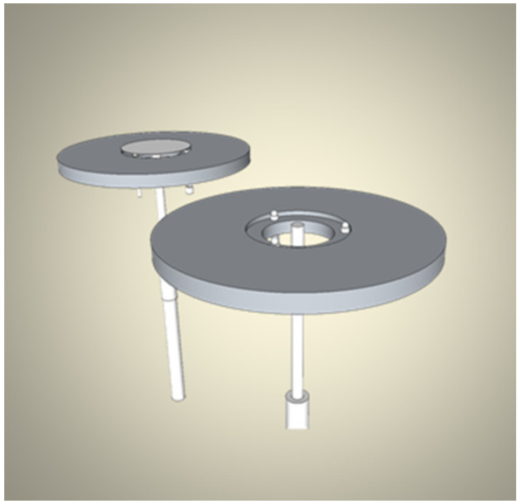
Patent granted:
US 9,601,391 B2



The prototype





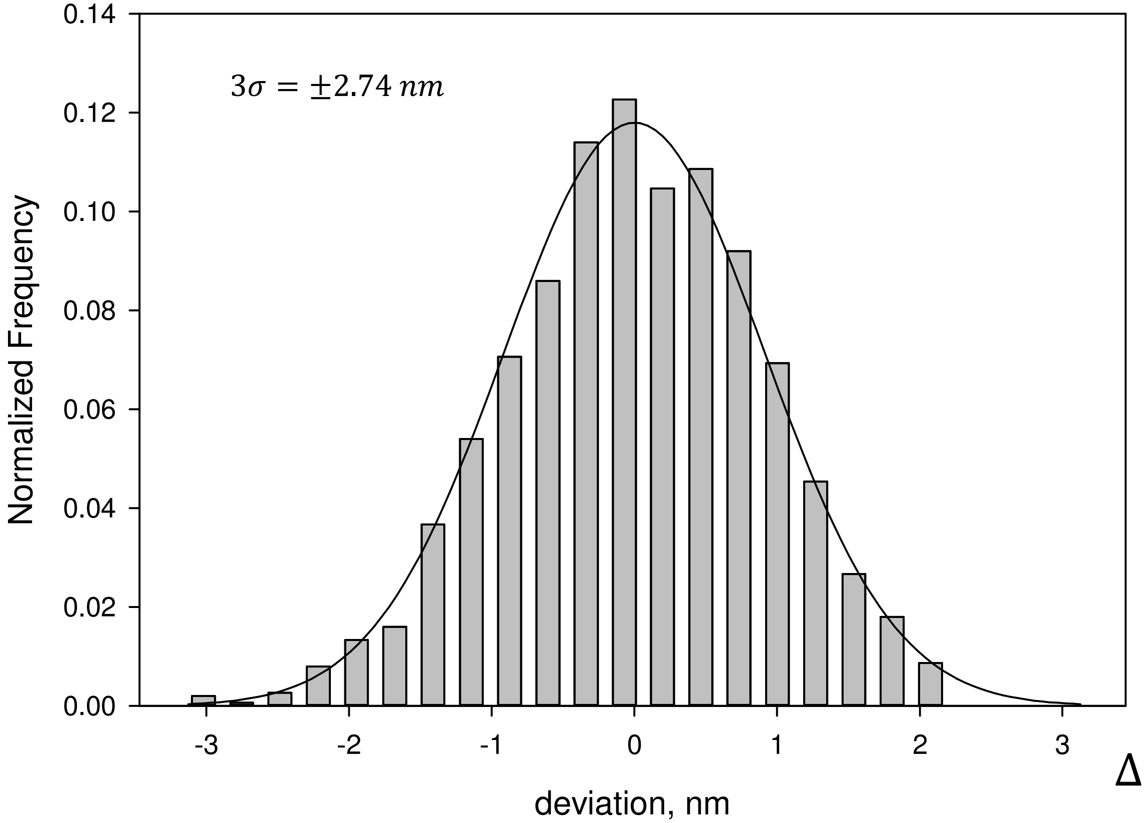


Stress Measurement Resolution (circular substrate):

Minimum force per unit width:

$$\Delta(\sigma h_f) = \frac{4}{3}\frac{E_s}{(1-\nu_s)}\left(\frac{h_s}{D_s}\right)^2 \Delta\delta$$

Fiber Optic Random Noise Distribution



Minimum force per unit width for 280 µm Si <111>, 1 in. dia. substrate:

$$\Delta(\sigma h_f) = 15\ MPa * nm$$

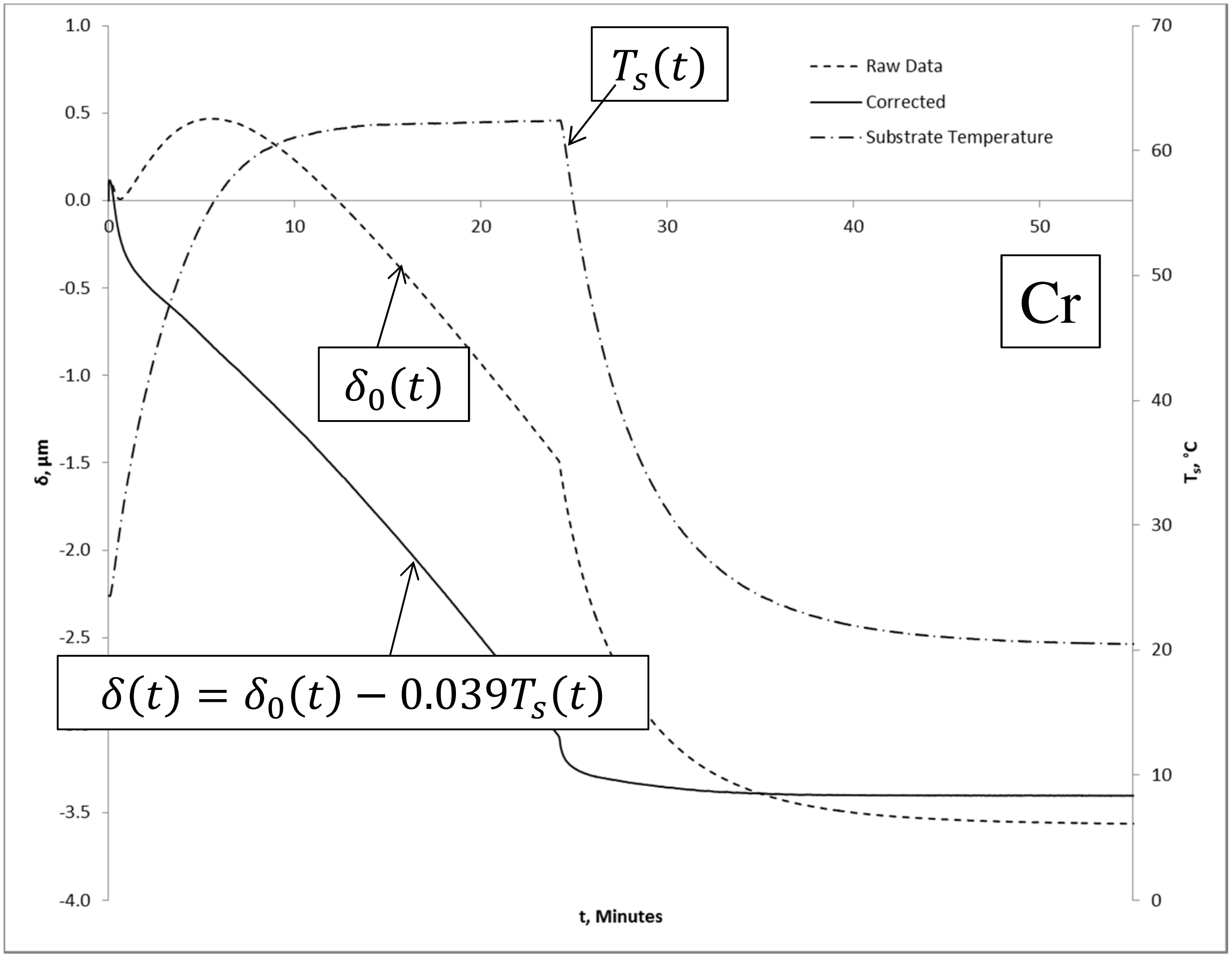
This means for example that:
A film with $\sigma_i = 100\ MPa \Rightarrow$
$\Delta h_f = 6.7\ nm$

For smaller values of stress, we can enhance the sensitivity by utilizing thinner substrates.

$$\sigma h_f = \left(\frac{h}{h_0}\right)^2 (\sigma h_f)_0$$

$\Delta h_f = 8.5$ Å for 100 µm substrate

Raw data must be corrected for thermal expansion error



$$\delta(t) = \delta_0(t) - 0.039T_s(t)$$

Measured data corrected for systematic thermal error

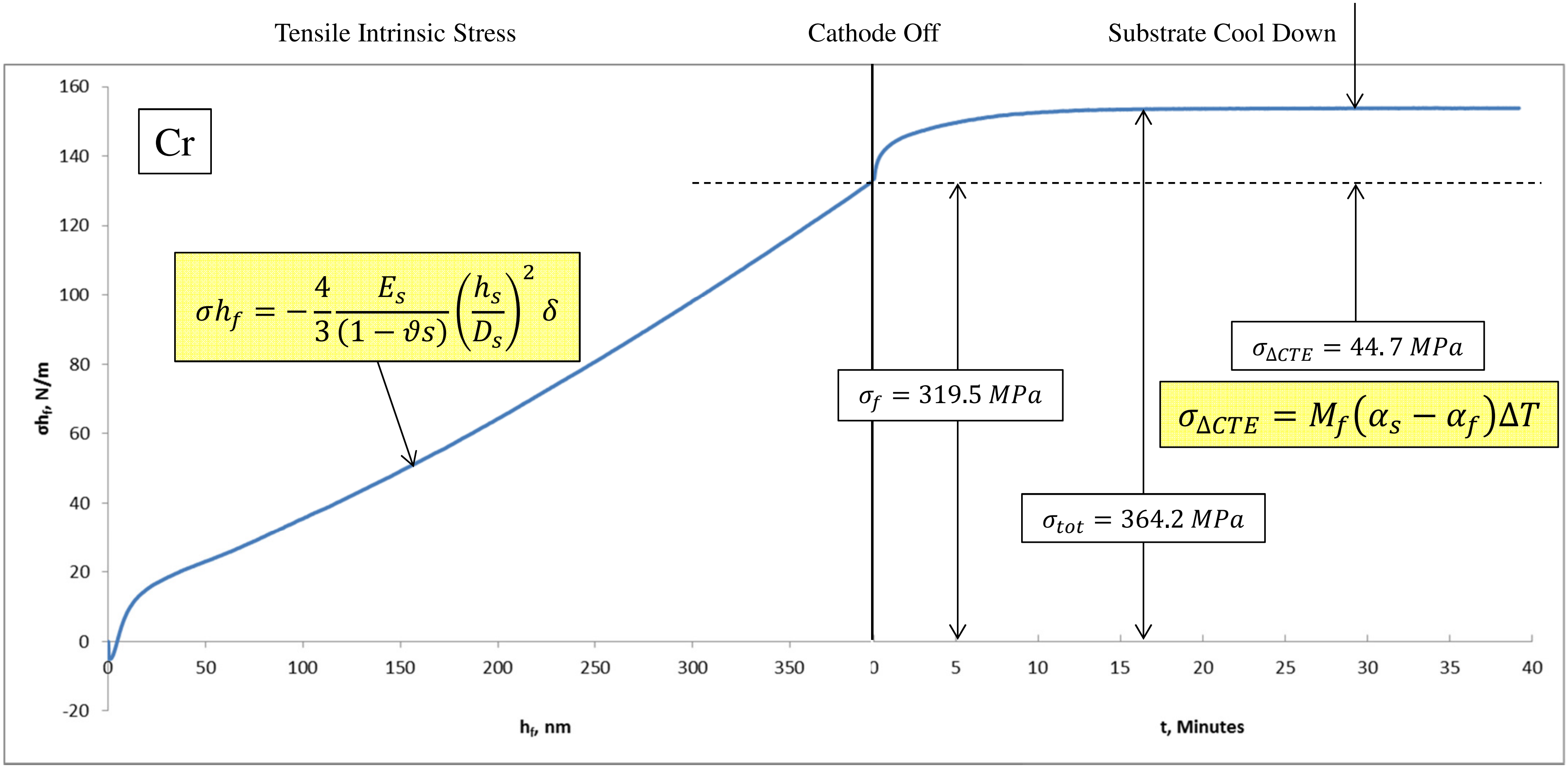
As a check of the results we can compare the measured contribution of the stress resulting from the mismatch in the thermal expansion coefficient between the film as substrate, $\sigma_{\Delta CTE}$,to the calculated value using bulk constants for chromium to:

With: $M_f = 279\ GPa,\ \alpha_f = 6.2 \times 10^{-6}\ °C^{-1}, \alpha_s = 2.2 \times 10^{-6}\ °C^{-1},\ \Delta T = 42\ °C$

$$\sigma_{\Delta CTE}^{calc} = 46.8\ MPa$$

$$\sigma_{\Delta CTE}^{measured} = 44.7\ MPa$$

Agreement to within 4.5%



$$\sigma h_f = -\frac{4}{3}\frac{E_s}{(1-\vartheta s)}\left(\frac{h_s}{D_s}\right)^2 \delta$$

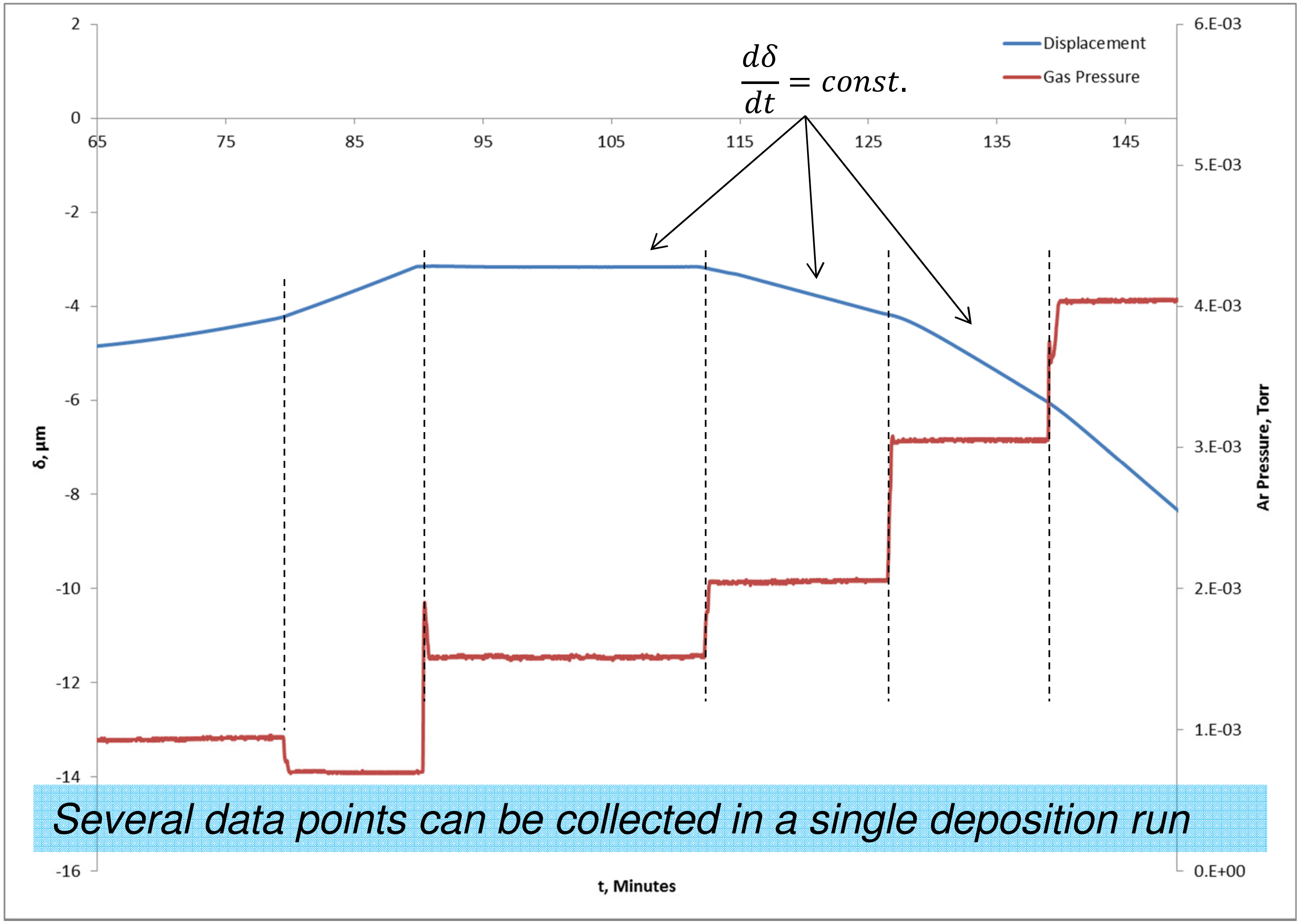
$$\sigma_f = 319.5\ MPa$$

$$\sigma_{\Delta CTE} = 44.7\ MPa$$

$$\sigma_{\Delta CTE} = M_f\left(\alpha_s - \alpha_f\right)\Delta T$$

$$\sigma_{tot} = 364.2\ MPa$$

Variation of instantaneous stress with Ar process pressure



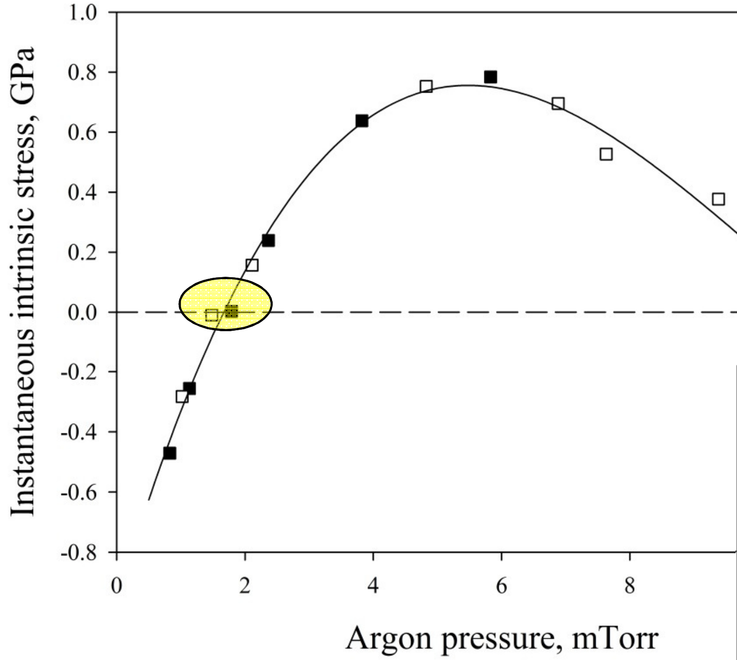
Several data points can be collected in a single deposition run

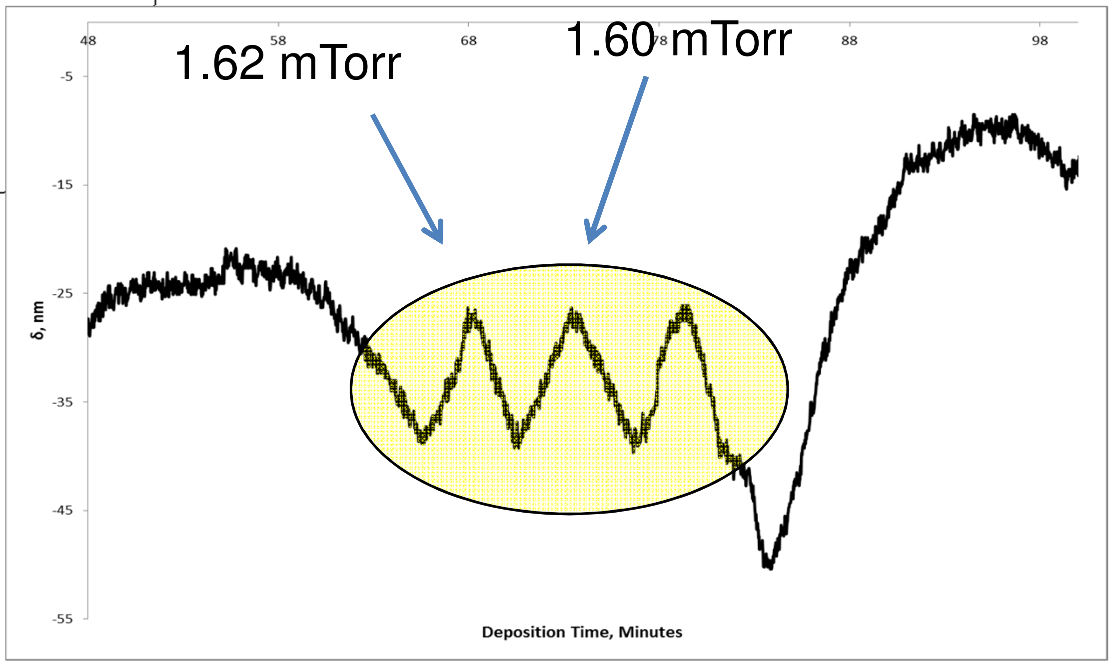
Stress reversal in metal films

$$\frac{d(\sigma h_f)}{dh_f} = -\frac{E_s}{3\xi(1-\vartheta s)}\left(\frac{h_s}{r}\right)^2 \frac{d\delta}{dt}$$

Results are consistent with D.W. Hoffman, Internal stress of sputtered Chromium, Thin Solid Films, 40 (1977) 355-363

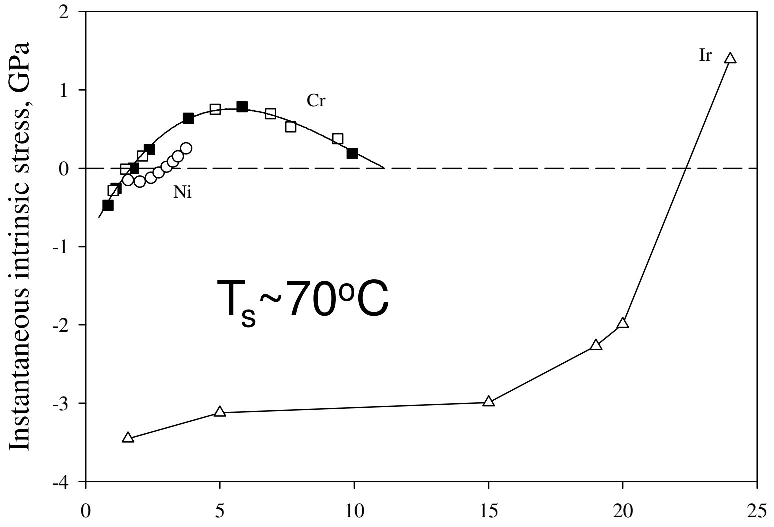
Stress reversal in Cr with argon pressure has been measured with the instrument. Consistent with the previous work of Hoffman (i.e. stress reversal).



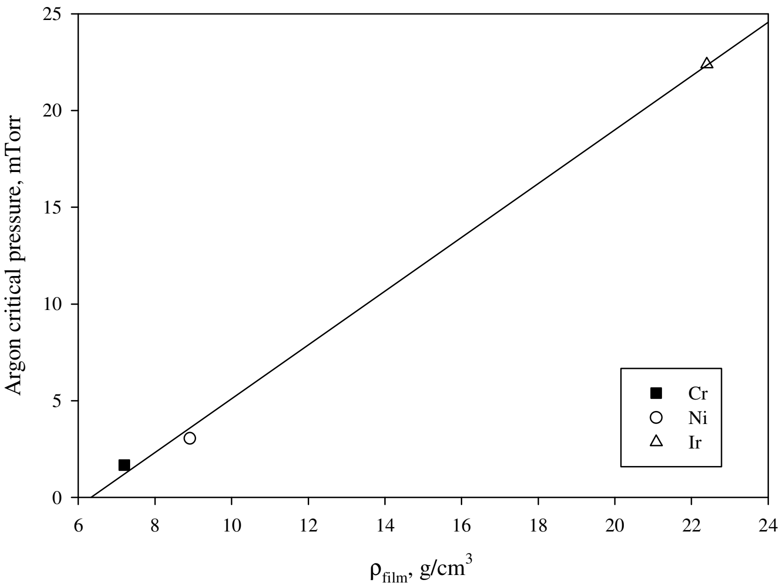


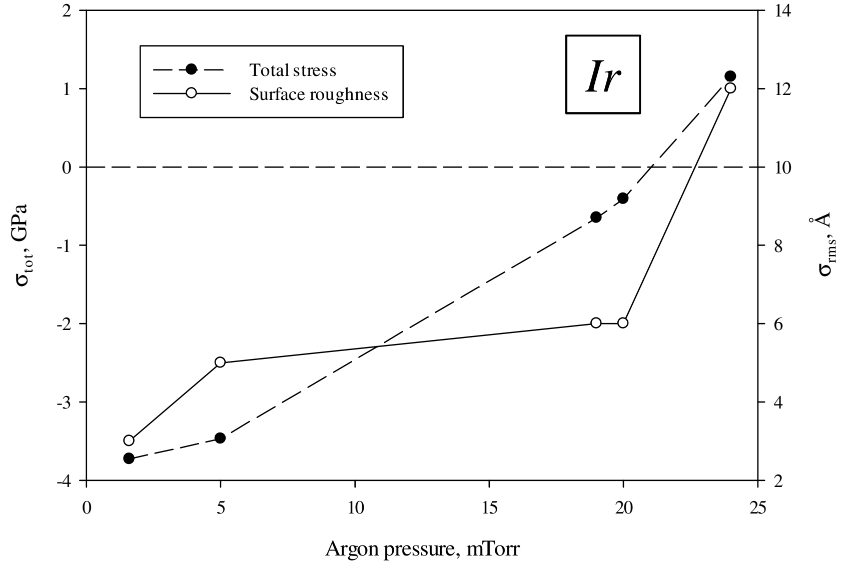
Measurement sensitivity is better than resolution in the control of Argon pressure.

Stress reversal cond't



Scaling of the critical pressure with film density

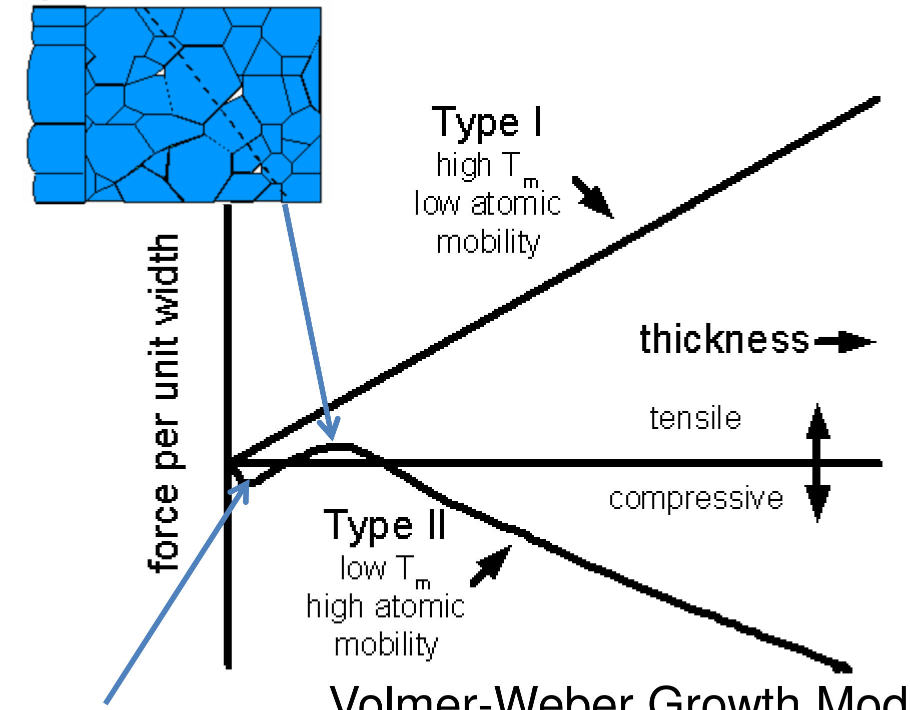




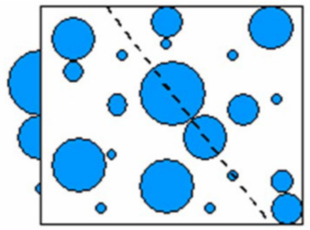
- Iridium exhibits stress reversal at high argon pressure (~22 mTorr)
- At this pressure the film's microstructure causes surface roughness that exceeds the tolerable limit of 4-5 Å for soft X-rays
- We can't use the stress reversal mechanism to achieve zero stress in Ir films

Film stress and microstructure

Island coalescence

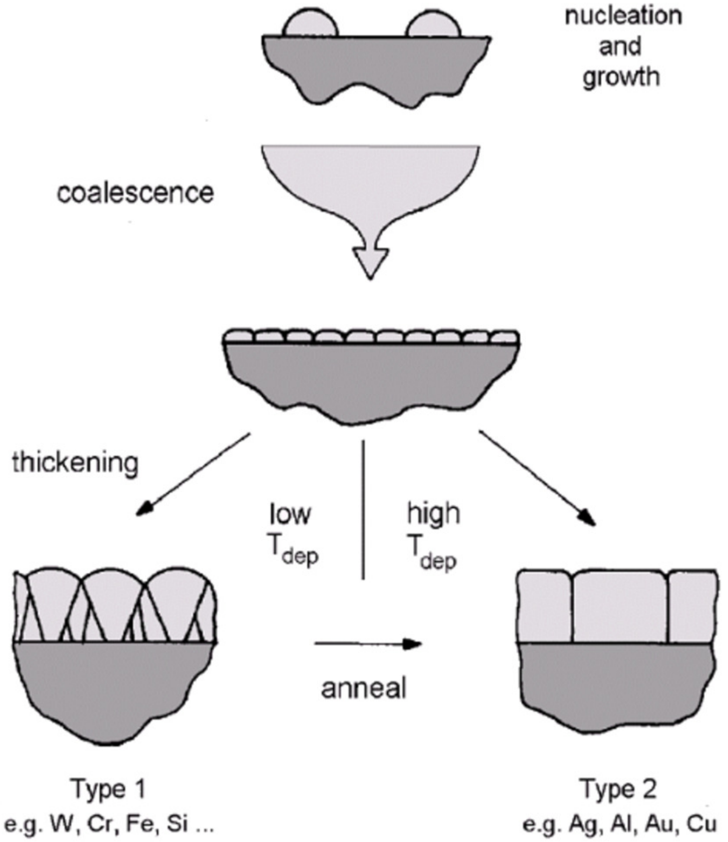


Volmer-Weber Growth Mode



Nucleation & island growth

<u>Depends on:</u>
Substrate temperature
Argon pressure
Mass of sputtered atoms
Substrate bias
Surface energy

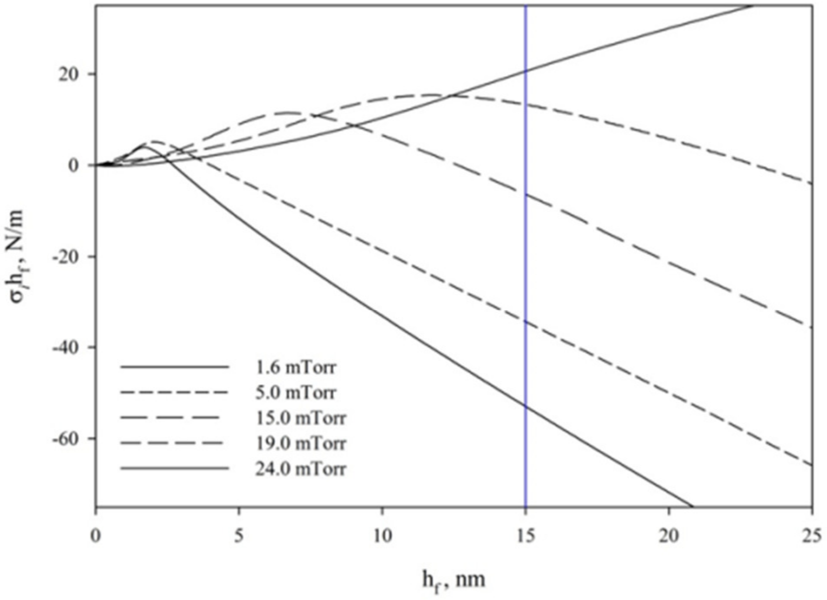


Surface roughness increases with film thickness

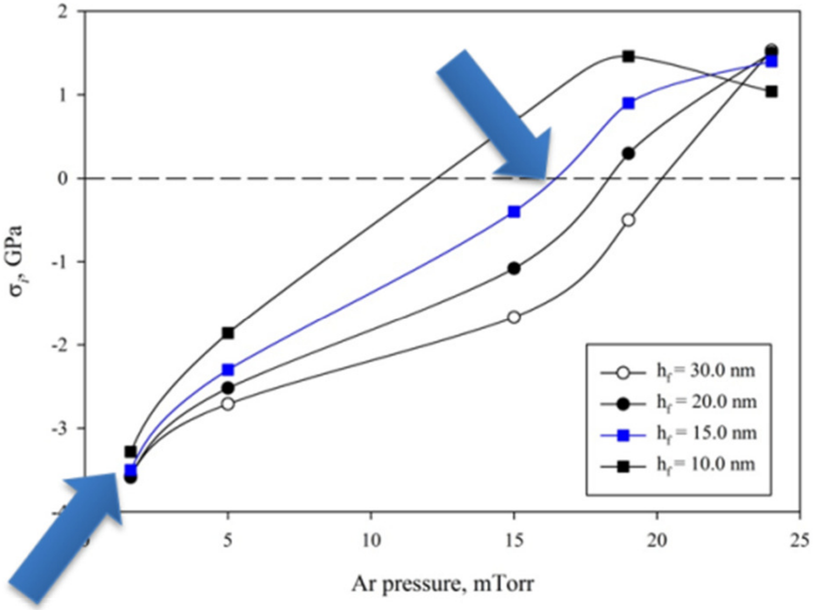
Low surface roughness

Stress in Iridium

Iridium instrinsic force per unit width

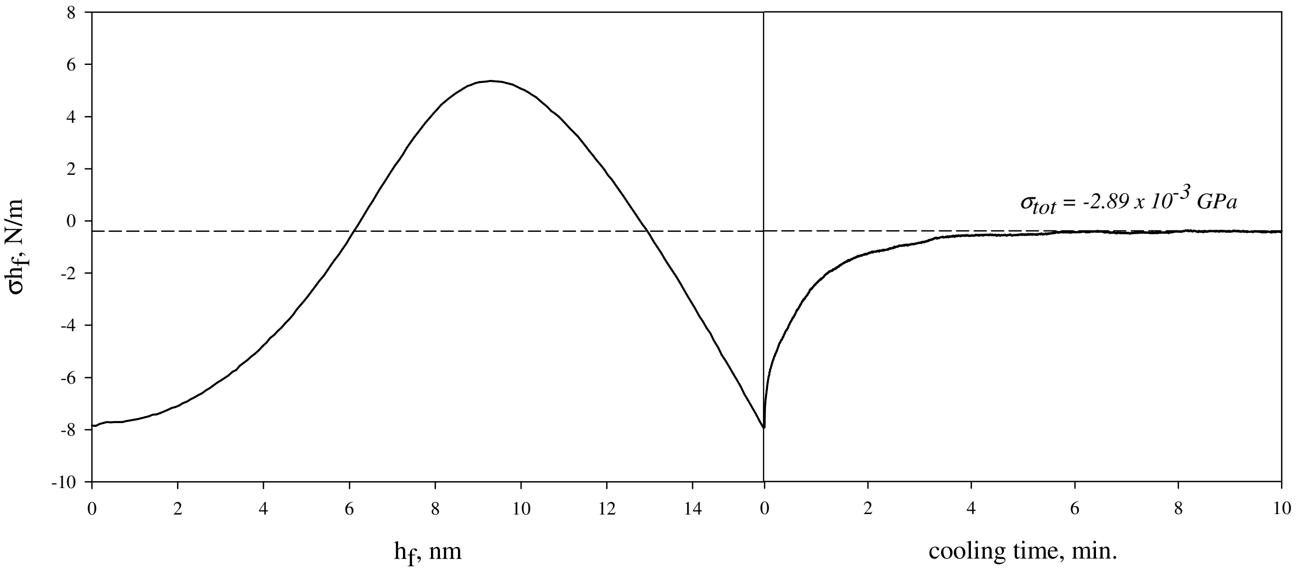


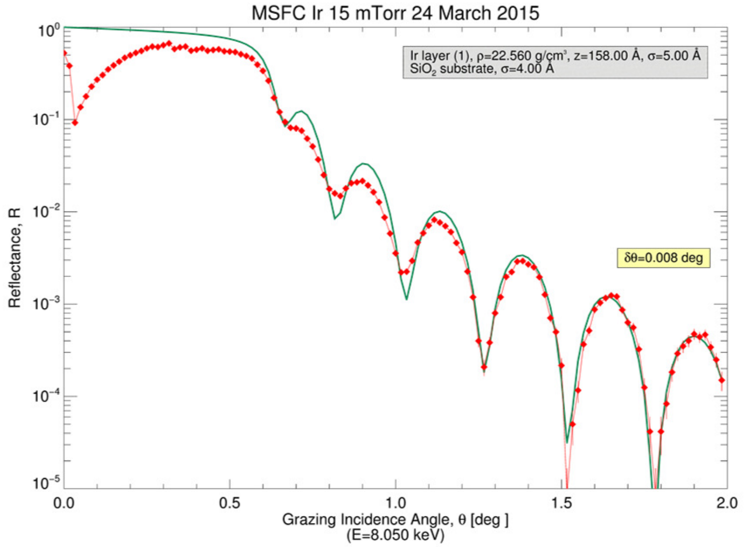
Zero stress @ 16.5 mTorr



-3.7 GPa

Near-zero stress in Iridium





Reduction in the total stress by 3 orders of magnitude (i.e. to -2.89 MPa)

Good adhesion

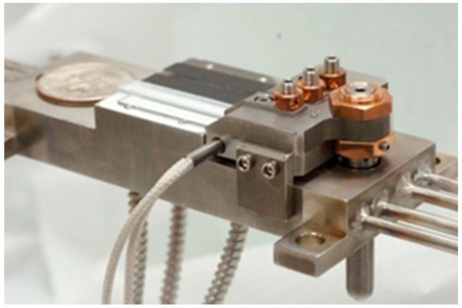
Promising result: 5Å rms roughness

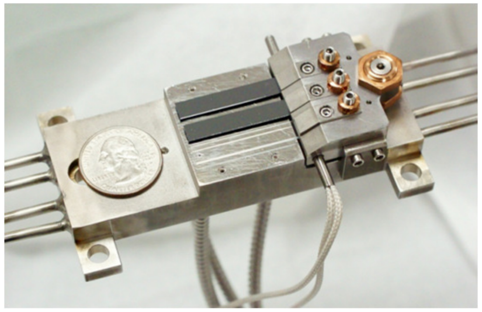
Further reduction in the roughness is possible through optimization of Ar pressure

Refined approach: Cantilever substrate

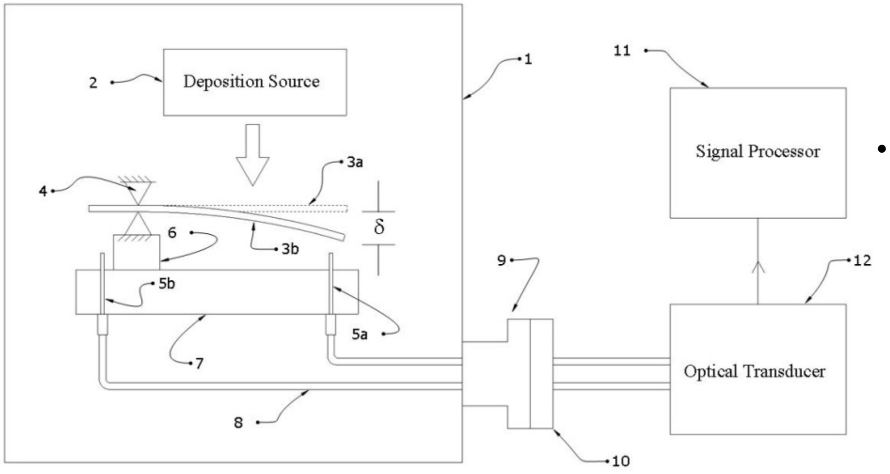
Stoney Eqn. for cantilever:

$$\sigma h_f = \frac{E_s h_s^2 \delta}{3(1-\nu_s)L^2}$$



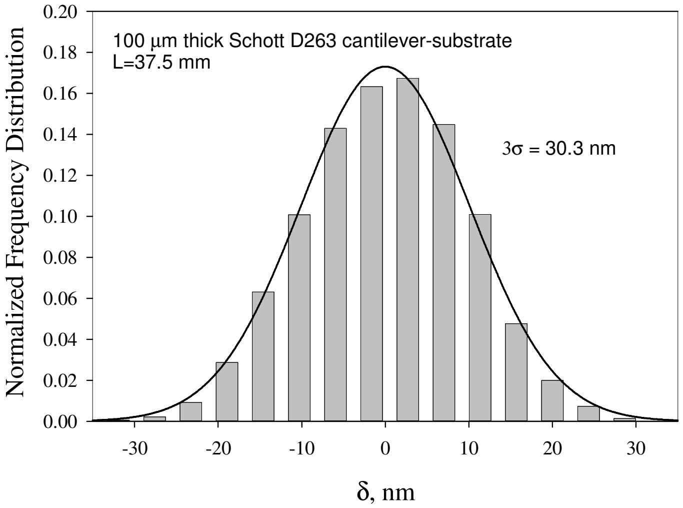


$$\Delta(\sigma h_f) = 9\ MPa * nm$$



- The use of cantilever substrates can theoretically provide a four fold increase in measurement sensitivity in comparison to circular substrates of a similar thickness and characteristic dimension (Ds~L)
- The vibrational noise, however, also increases resulting only in a factor of ~1.7 improvement.

D.M. Broadway, U.S. Patent 9,601,391 (Granted March 2017).
D.M. Broadway, U.S. Patent Application 15/425,740 (Filed February 2017).

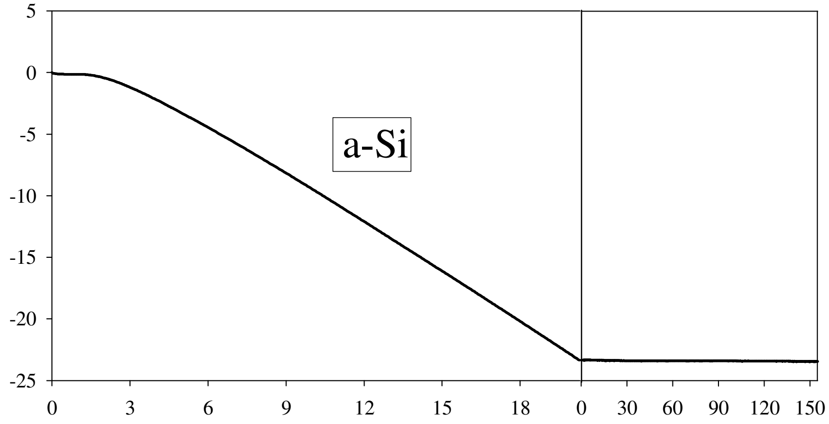


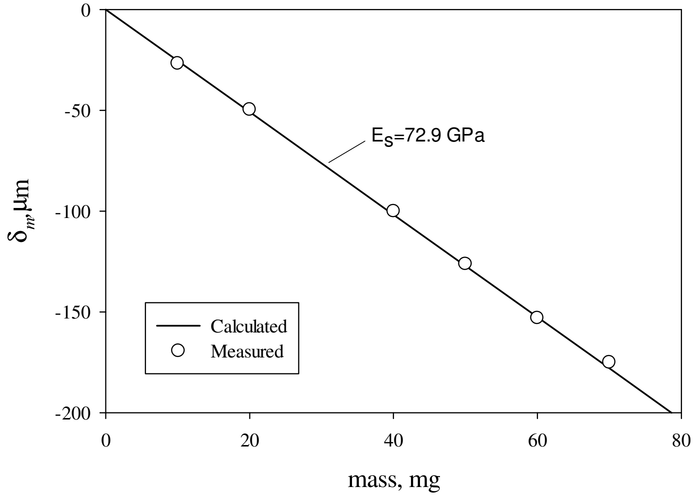
In-situ stress of single layer thin films

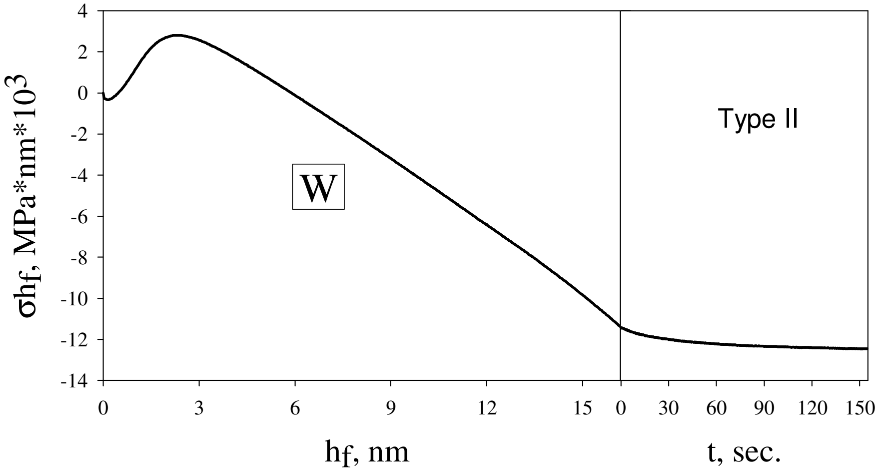
T_s~27-30°C, 2.5 mTorr Ar
100µm thick Schott D263

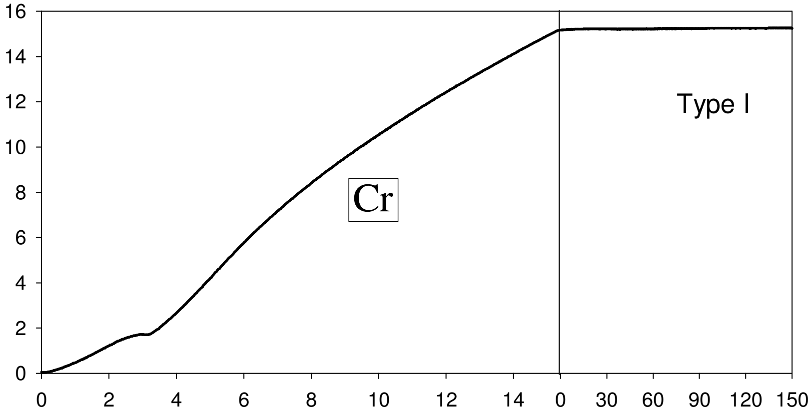
Calibration masses placed on cantilever tip used to validate substrate modulus and linear range of the sensor:

$$\delta_m = \frac{2mgx^2}{E_s b h_s^3}(3L - x)$$

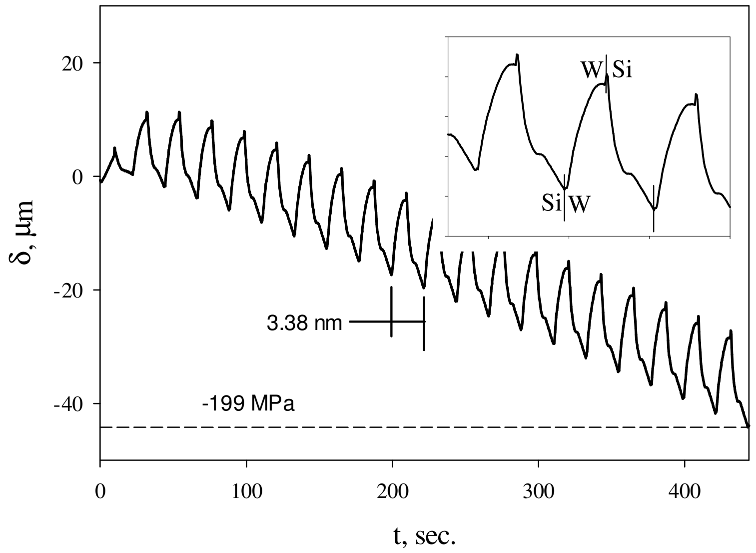


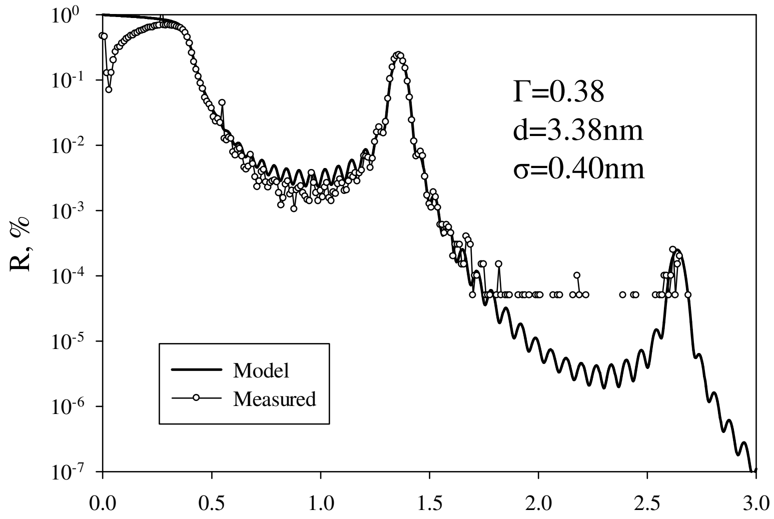






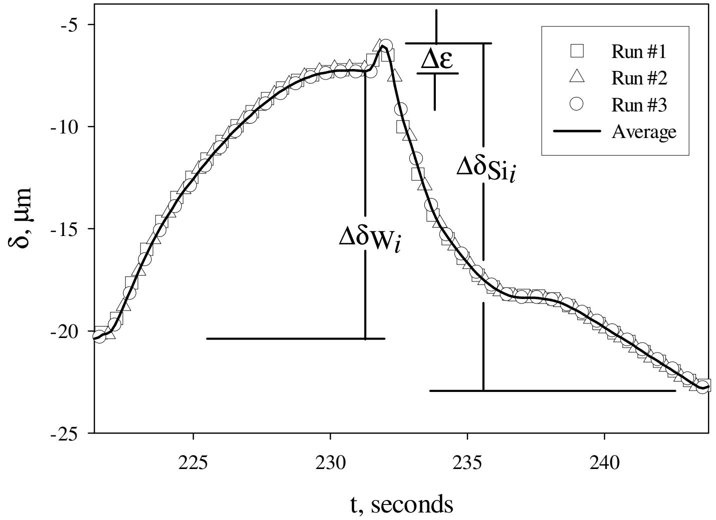
Device performance

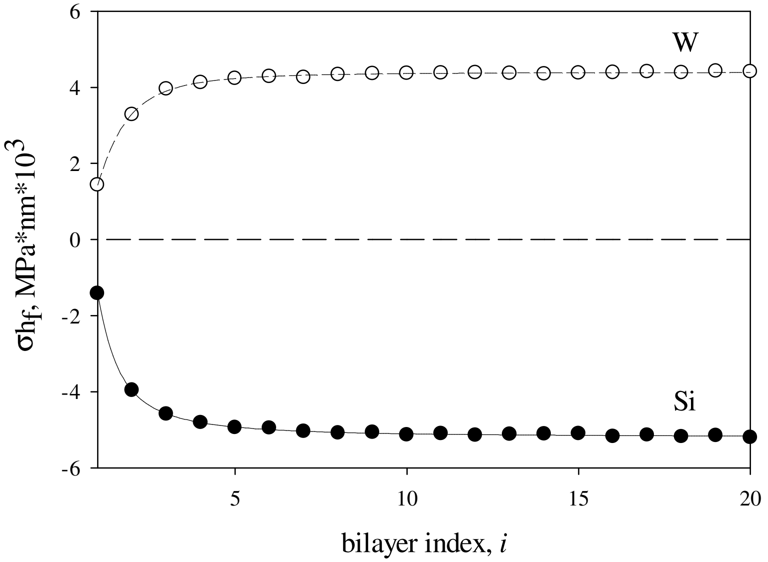




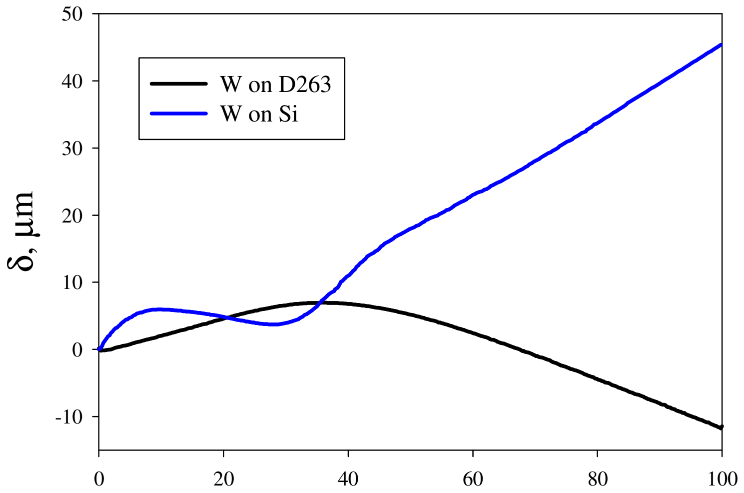
±0.5% within run repeatability

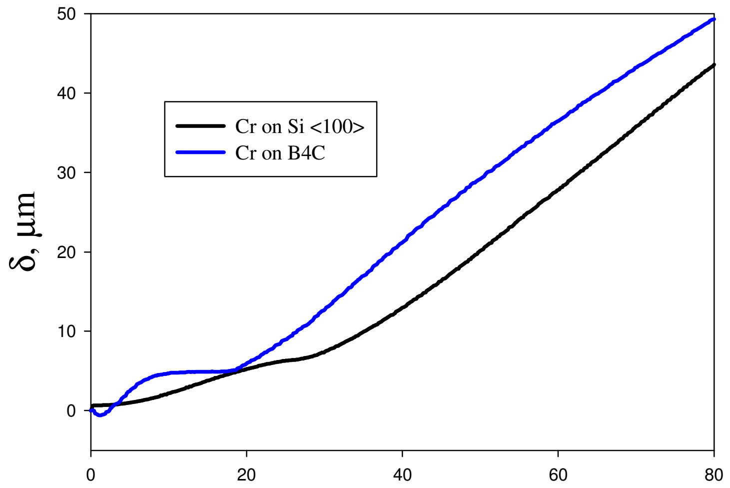
±2.5 % run-to-run repeatability

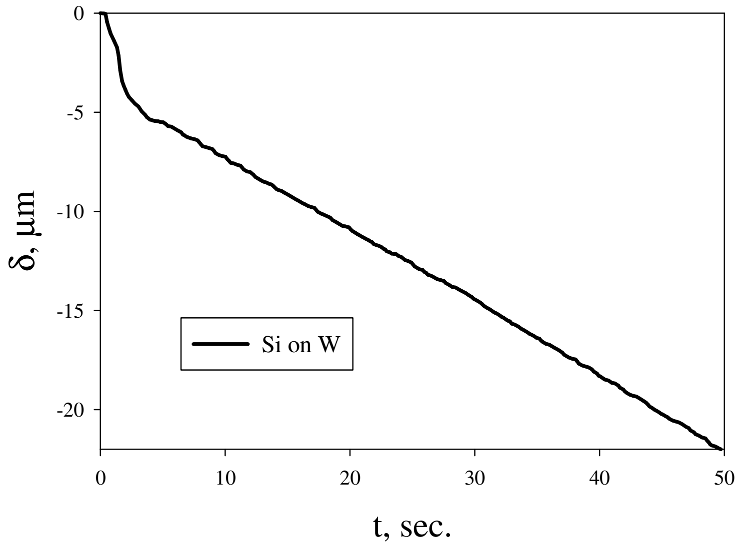


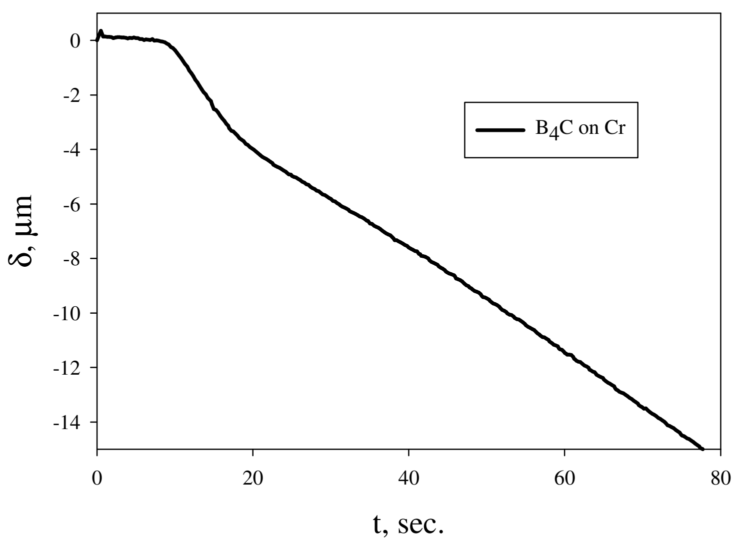


Effect of material interfaces on the film stress (W, Cr-based)



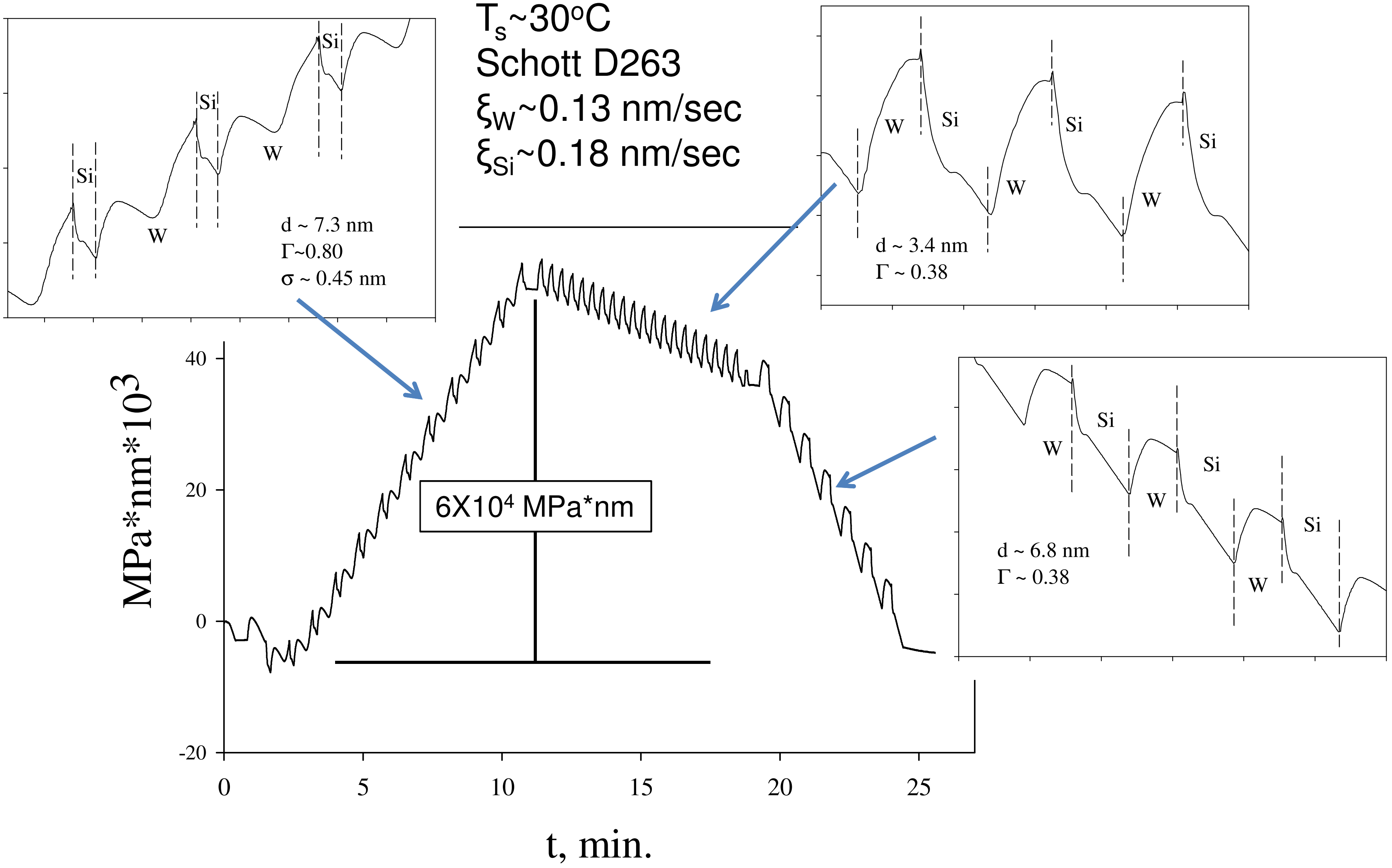




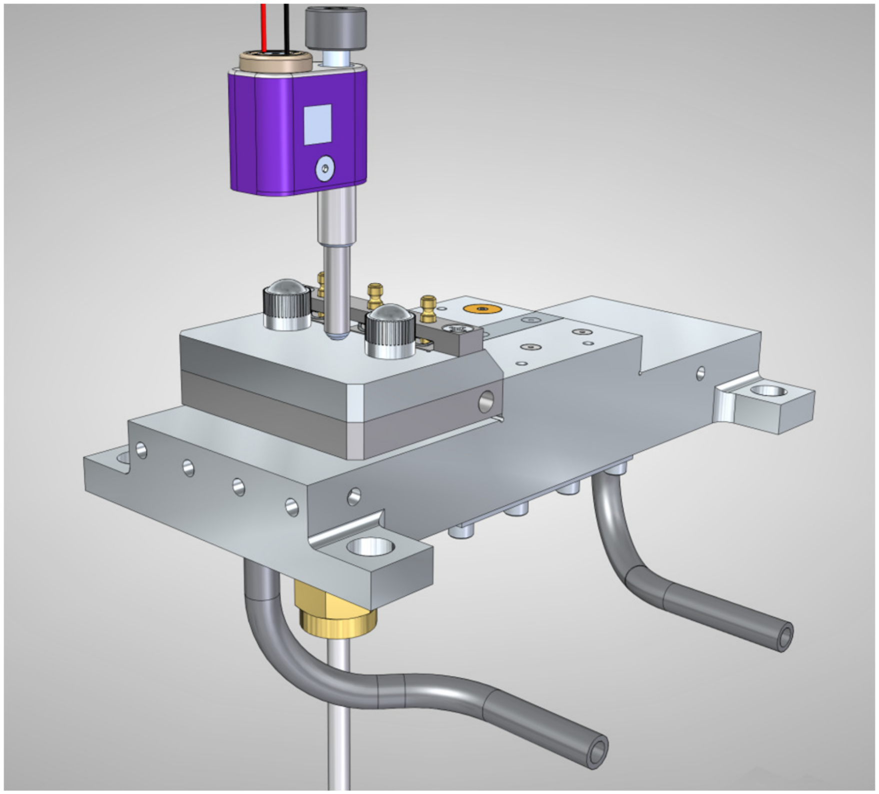


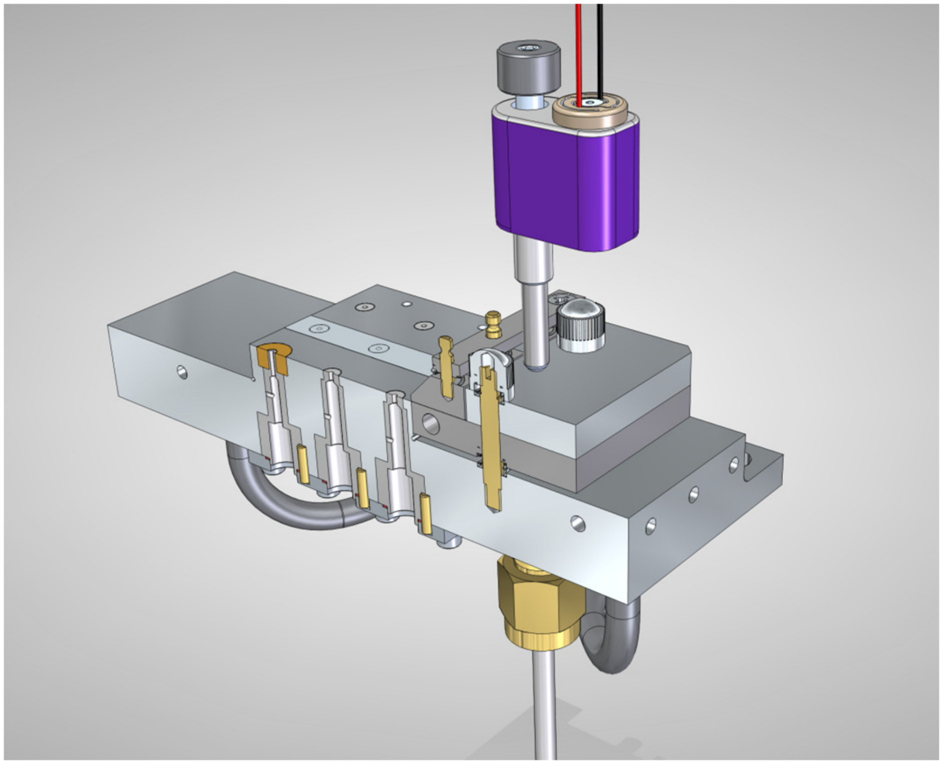
In-situ stress in W/Si multilayers

T_s~30°C
Schott D263
ξ_W~0.13 nm/sec
ξ_{Si}~0.18 nm/sec



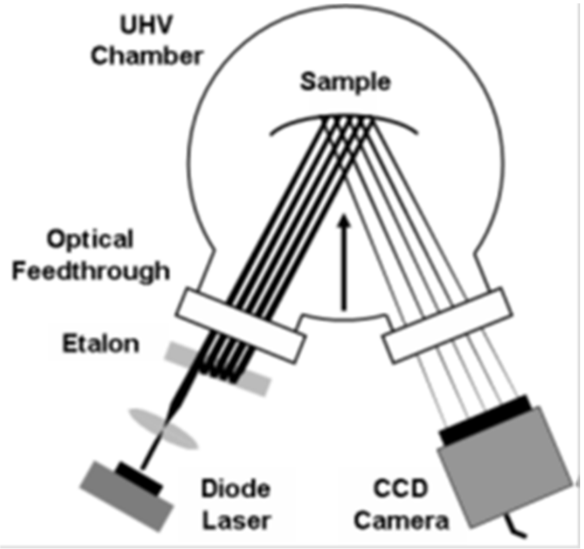
Second refinement with advanced capability (ongoing)



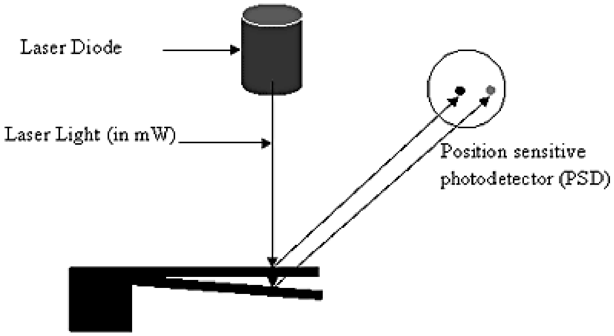


Comparison to state-of-the-art stress measurement:

Multi-beam stress sensor (MOSS):



Micro cantilever:



Minimum detectable stress $\Delta\sigma h_f$:

- Ranges from 0.5-50 MPa*nm depending on method and substrate (i.e. geometry and mechanical properties)
- MOSS is 50 MPa*nm for 100 µm thick silicon substrate

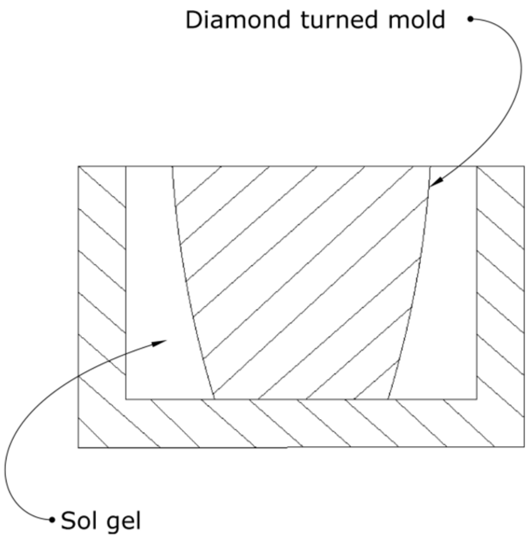
Draw backs with current optical methods:

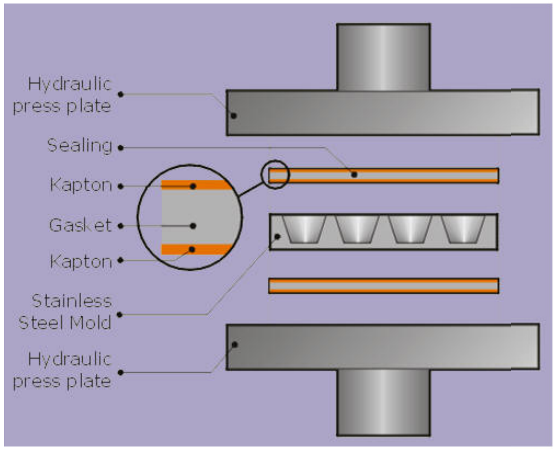
- Requires external optical access to the substrate through angled viewports
- Limited to specific deposition geometries
- Complex
- Requires the use of opaque substrates such as crystalline silicon.
- Film side is measured which can result in destructive interference effects when measuring transparent films.

Replicated silica aerogels for ultra lightweight mirrors

Rapid supercritical extraction method (RSCE)

Partner: Union College NY



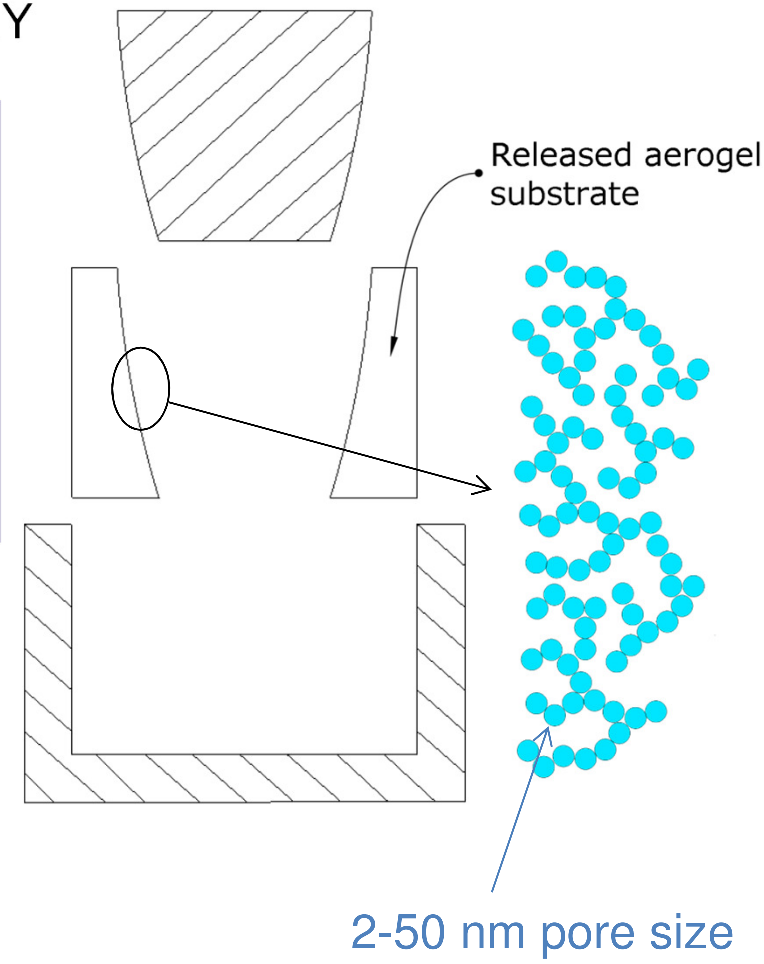


Mixture of precursors and nanoparticles (sol gel)

The gel is a colloidal systems in which a nanostructured network of interconnected particles spans the volume of the liquid.

If the liquid in the gel is simply allowed to evaporate, capillary stress would collapse the gel's delicate solid framework. Therefore, a process of supercritical drying is used.

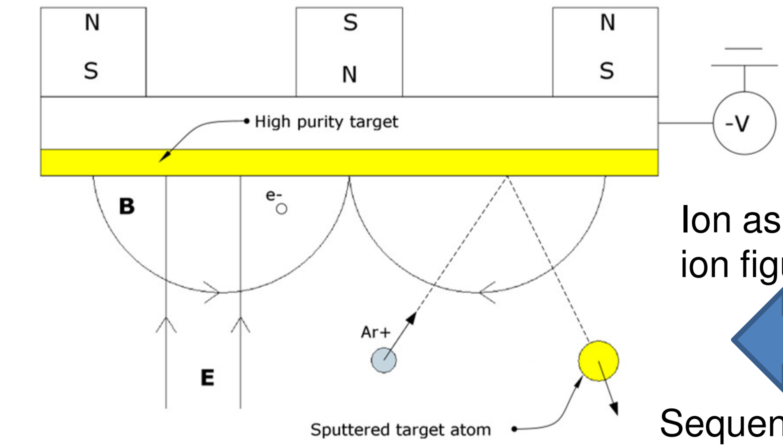
An autoclave is used to heat/pressurize the liquid past its critical point where it is transformed into a supercritical fluid. The supercritical fluid loses all surface tension and can no longer exert capillary stress. Isothermal depressurization at the critical temperature is then used to transform the supercritical fluid to a gas.

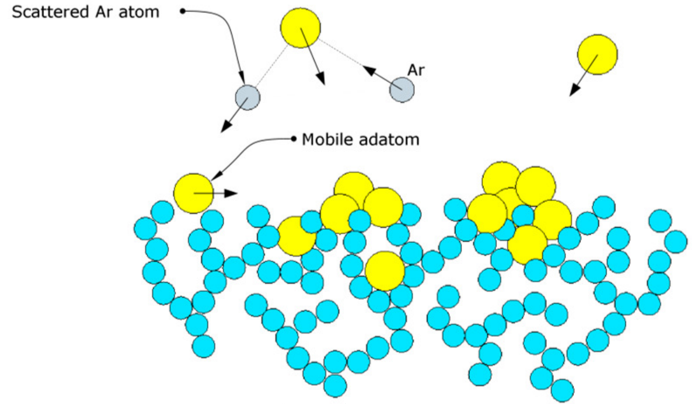


Dry, low-density, porous, solid framework of the gel. Typically
95-99% air/vacuum by volume.

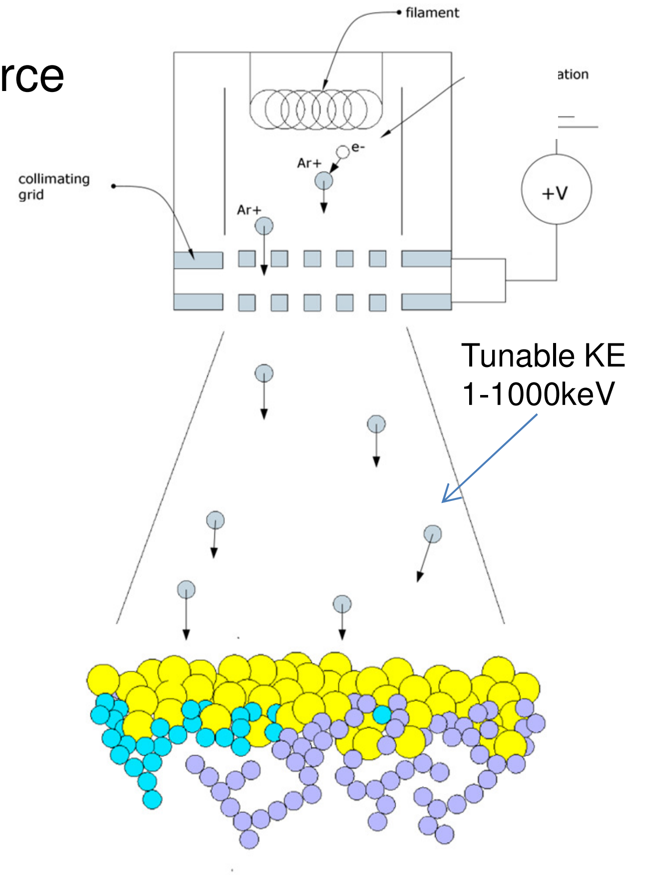
Planarization, optical coating of areogels

Magnetron Sputtering





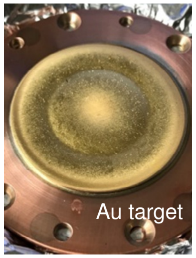
Ion Source



Tunable KE
1-1000keV

Ion assisted deposition,
ion figuring (optional)

Sequentially or concurrently



Process conditions

- Gas pressure
- Temperature
- Mass of sputtered species

Adatom mobility

Film microstructure
Film stress

Surface
roughness

Optical performance

- Specular reflectivity
- Light Scattering
  - Imaging Resolution

Tolerable micro roughness, σ, increases with wavelength of incident radiation: σ< 3-5 Å for x-rays
< 1-3 nm for visible light

Thank you!